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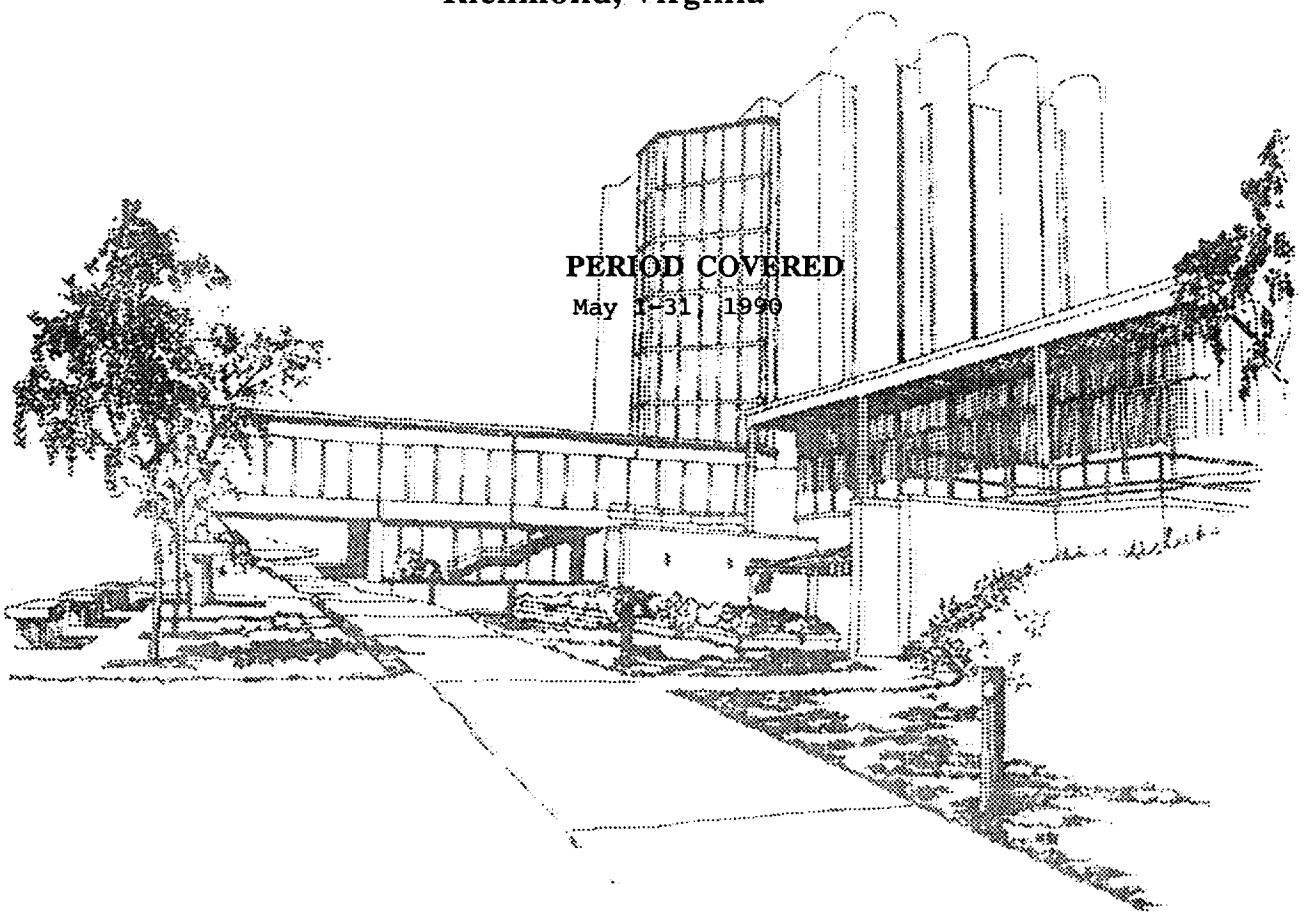

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May 1-31, 1990

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*N/R = No Report

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PROJECT NUMBER: 2108
PROJECT TITLE: New Product and Filter Technology
PROJECT LEADER: W. T. Callaham
PERIOD COVERED: May, 1990

I. SAUNA

A. **Objective:** Develop products to compete against Barclay utilizing a novel filter system.

B. **Status:** Three prototypes for the GCC area (4mg tar) were subjectively evaluated by Flavor Development versus a Saudi Barclay. Two models (dual concentric & 1.6/41000 CA; and mono paper) achieved parity while the third model (dual SCS-III & 1.6/41000) was preferred. Three earlier prototypes were remade to adjust deliveries and are currently being analyzed. These have filter systems of dual concentric & carbon-on-1.6/41000 CA, dual 1.6/41000 CA & paper, and dual Astra & 1.6/41000 CA. The best candidates will be forwarded to EEMA for evaluation.

Also, two models for Japan (5mg tar) were subjectively evaluated versus a Japanese Barclay and both were preferred. These had filter systems of dual concentric & carbon-on-1.6/41000 CA; and dual SCS-III & carbon-on-2.6/42000 CA. These have been submitted to Japan Product Development for evaluation.

C. **Plans:** 1. Finalize construction of the GCC model submit for EEMA approval.
2. Finalize construction of the Japanese model and test on Danchi panel.

II. ASH TRAY ODOR

A. **Objective:** Develop products which facilitate the elimination of ash tray odor.

B. **Status:** As a result of encouragement over the most recently produced odor-less ash tray, fabrication was begun by an outside machine shop for additional models. Delivery is expected by early June.

C. **Plans:** Complete fabrication of additional models.

III. MENTHOLATION TECHNOLOGY

A. **Objective:** To develop alternative mentholation techniques which offer a product or processing advantage.

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B. **Status:** A six-week aging study was completed of cigarettes mentholated via PG/menthol-on-foil (20%/80%). Menthol-in-smoke levels remained at approximately 0.5mg throughout the test period. Equilibration between the inner and outer cigarettes took approximately 3 to 4 weeks. Subjectively, only minor differences were noted from the control MOF model. SOLVIC spotting analysis after 6 weeks indicated more than twice as many spots for the test cigarettes as for the control. A similar trial is in progress with cigarettes mentholated via slightly different solutions to address the above concerns. These solutions are 25% PG/75% menthol and 15% PG/80% menthol/5% ethanol.

A brief factory trial (limited due to production requirements) of pre-embossed 30-lb mentholated foil was conducted in Louisville to demonstrate machinability. Marlboro Lights 100 cigarettes were packed without any foil curling or breakage. Another trial has been requested for more extensive sampling.

Analysis of a menthol/β-cyclodextrin complex prepared in-house indicated a menthol content of 9.7% versus 8.8% for a similarly prepared vendor sample. Product applications are currently being pursued.

A meeting was held with Engineering to review the results of the mentholated insert study and to request further economic evaluation.

A study was initiated as a result of work done by Eastman on improved menthol stability using unplasticized filters.

C. **Plans:** 1. Complete a factory trial of foil embossing prior to mentholation.
2. Complete the menthol/PG-on-foil study.
3. Continue development of an encapsulated menthol.
4. Complete a study of acidified filters on menthol response.
5. Complete the unplasticized filter study.

IV. WEB-TYPE FILTER MATERIALS

A. **Objective:** To characterize web-type filter materials and develop subjectively acceptable products with them.

B. **Status:**

Paper Filters: MF Lights Menthol 100 models were prepared with mentholated paper filters and submitted to CTSD for analysis as part of a study to investigate their subjective and menthol delivery performance.

A process/product comparison of our web filter maker and Intertaba's filter maker was completed . All data from this study is being summarized and a report being written.

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Two bobbins of Tela crepe paper were received with 2% and 20% creping levels. The materials were evaluated to determine the effect of creping on filter quality and web runnability. Both materials ran well, however, different machine conditions were required for the two materials. The creping appears to allow more flexibility in runnability which would be very important for high speed filter making.

A filter tip RTD gain study was completed. RTD increases were not observed as previously seen between web filter rods and finished filter tips. The study will be replicated on different equipment.

PM Web: Sample webs were produced at the University of Maine using 75% 1/8" cut CA staple and 25% softwood fiber. Various refining levels were evaluated and CMC and PVA binders were used to improve elongation. Creping of the web at two different machine positions was unsuccessful. The sample bobbins are currently being evaluated for filter making machinability and cigarettes will be produced for analytical and subjective evaluation.

Paper/Pulp Acetylation: Filter rods were handmade from hand sheets comprised of 75% acetylated wood pulp + 25% softwood pulp. In addition, filter rods were machine made from a 30 ft. sample of acetylated cellulose web from Physical Research. These two acetylated cellulose filter models are being put into cigarette models for preliminary subjective screening.

CA Web: Samples of the most recent Kimberly-Clark produced CA Web is currently being characterized.

A corrugation correlation study was conducted with Celanese using "sister" bobbins. Data is currently being analyzed.

Sample filters of Dexter produced CA Web were received from Celanese for analytical and subjective characterization. One model has a modified recipe containing 10% cellulose in place of CA staple.

C. Plans: 1. Complete paper filter additives testing.
2. Complete mentholated filter study.
3. Continue development of PM web, acetylated paper, and coated papers.
4. Complete the evaluation of Celanese CA web.
5. Investigate polymeric webs.
6. Complete the filter tip RTD gain study.

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IV. NOVEL VENDOR FILTERS

A. Objective: To investigate vendor filters and filter materials for potential applications where increased efficiency, taste response or a novelty is needed.

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B. Status:

Concentric Filters: Mega look-a-like test (concentric) and control (CA mono) models were made with MFUL filler and subjectively evaluated by Flavor Development. The control had slightly increased impact and was more peppery than the test. FTR-produced Mega cigarettes with CA mono and concentric filters are currently being tested.

Rhodia Supercrimp Filters: Subjective evaluation of Rhodia 2.1/25000 supercrimp CA tow by Flavor Development personnel indicated no difference from FT-107 or FT-222 at equal RTD and delivery. This tow item allows for approximately 25mg per filter tip weight reduction over the above controls. A final report is being prepared.

Three different filter systems are currently being evaluated as part of a smoke presentation study. The filters are an SCS-III, Astra and CA control.

Project 605 models with SCS-III filters, hole-in and hole-out, were subjectively evaluated by Flavor Development. The SCS filter with the hole toward the smoker imparts a higher impact while holding filtration efficiency constant.

C. Plans: 1. Complete evaluation of the Mega filter.
2. Complete the smoke presentation study.
3. Provide specialty filters to others as requested.
4. Complete a study of MPF filters.
5. Complete the dual cambridge pad holder evaluation.

V. PRODUCT DEVELOPMENT SUPPORT

A. Objective: To provide cigarette/filter design, modelling and testing support for major new product development programs.

B. Status:

Ambrosia: Samples of entrapped vanillin were prepared for testing by Flavor Development. One sample contained about 10% in β-cyclodextrin and the other contained about 50% in Ca-adipate.

Best of the Lowest: A request has been submitted to semi-works for a 1mg BOLD model using a dual UHF/CA filter (18/9mm configuration).

A request was submitted for 1mg BOLD cigarettes using recessed end filters.

Concentric filters for a 1mg Bold model were designed and are being made by Filtrona. These concentrics have either a paper core or paper periphery in parallel

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with 1.6/41000 tow. SCS-III filters for a 100mm version have also been designed and ordered.

Machinery Development: A modified ESP machine for Superslims papers was received and is being installed in the Filter Development Lab.

C. **Plans:** 1. Continue development for new products as required.
2. Complete ESP unit installation.

VI. CARBON STUDIES

A. **Objective:** Conduct fundamental studies of carbons and carbon filtered cigarettes to better understand their performance.

B. **Status:** Analytical Research, using TGA methods, completed a study of our carbons types taken from Lark Milds, PM Lights and Parliament 100's cigarettes at several aging intervals. The data indicate that the carbon in all three carbon cigarettes is absorbing propylene glycol and triacetin. The rate and amount absorbed varies among the three filter/carbon types. This data will be used to fingerprint the performance of the carbons for comparisons to new carbons.

Variability characterization of our three production carbons is in progress. Carbon tetrachloride activity testing was completed by Envirotrol and sieve/moisture testing is being completed by Incoming Materials QA.

Work has been initiated on identifying and characterizing competitive product carbons. Carbon was taken from Cabin Milds filters and forwarded to Analytical Research for determination of particle size and metal content.

C. **Plans:** 1. Complete the coal vs. coconut study.
2. Evaluate the effect of carbon OV, sieve size, and activity on subjective response and recommend the optimal combination.
3. Complete the evaluation of incoming carbon variability.

VII. ALTERNATIVE PLASTICIZERS

A. **Objective:** Qualify alternative plasticizers which offer a product advantage.

B. **Status:** Subjective evaluations by a Flavor Development Panel were completed on PM Super Lights made with triacetin (control) and triethyl citrate (test) plasticizers. Carbon loadings of 40 and 52 mg were used. Overall, minimal differences were observed between the control and the test samples.

C. **Plans:** 1. Complete testing as planned and continue investigations of other plasticizers.

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PROJECT NUMBER: 2305
PROJECT TITLE: Applied Flavor Investigation
PROJECT LEADER: J. W. Swain
PERIOD COVERED: May, 1990

I. PROJECT ART:

ART-By-Product Utilization:

A. **Objective:** To evaluate and develop process modifications for the utilization of ART process by-product tobaccos.

B. **Status:** POL 3637 (Marlboro control vs. Marlboro with 2% MPC ART stems in RCB) was shipped and will close out the week of June 7, 1990. Since 2% double-batched citric acid stems in Pilot RL and 5% in RCB were subjectively comparable to their respective single-batch tests, combinations of RL and RCB with double-batched stems in Marlboro have been requested.

Initial 100% RL and RLB cigarettes have been made to determine the subjective influences of citric acid, CO_2 ART extraction cycle and various combinations. Analytical differences due to the specific processing stages will be compared to differences in subjective attributes in attempts to modify subjective attributes of ART stems.

C. **Plans:**

Complete Initial Tests of Combinations of Double-Batched Stems in RL and RCB	June, 1990
Evaluate ART Stem Attributes Due to Process Stages	June, 1990

II. OPERATIONS SUPPORT:

PROJECT GRAIN:

A. **Objective:** To significantly reduce alcohol levels in PM flavor systems, while maintaining product subjective integrity.

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B. **Status:** Further Flavor Development panel tests of Cambridge cigarettes showed no obvious differences for control vs. sonolated test aftercut with a 30% reduction in alcohol. A POL test has been requested to confirm the results on Cambridge cigarettes.

Subjective evaluations are in progress on Cambridge aftercuts to test a range of alcohol reductions up to 40% without addition of water to compensate for volume.

C. **Plans:**

Initiate POL Test of Cambridge Model	June, 1990
Complete Small-Scale Test of Marlboro Aftercuts	June, 1990

III. PARK 500 EVAPORATOR UPGRADE:

A. **Objective:** To evaluate and qualify the use of new evaporator system for RL's at Park 500.

B. **Status:** Startup of the new evaporator system on Line 3 at Park 500 is scheduled for mid-June. Plans are in progress to make control and test Marlboro fillers with RL's from Park 500 at the MC for POL 3634. A solubles level study is in progress to determine the subjective influences of higher solubles to be available from the new system.

C. **Plans:**

Complete Solubles Level Tests	June, 1990
Ship POL 3634	July, 1990

IV. DRY FLAVOR REPLACEMENT:

A. **Objective:** To develop, evaluate and establish specifications for dry flavor replacements.

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B. **Status:** Pack codes have been assigned for a POL test of control versus dry flavor replacement test RLB in Marlboro. Primary blends have been completed with cigarettes to be tested on MC panel prior to release for POL.

Alternate vendor samples of RSJB continue to be evaluated to qualify a second source. Pilot RLB trials were rescheduled for next month.

C. **Plans:**

Complete MC panel testing	June, 1990
Release POL cigarettes	July, 1990

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PROJECT NUMBER: 2306
PROJECT TITLE: Marlboro Standardization/International Support
PROJECT LEADER: W. R. Bell
PERIOD COVERED: May, 1990

I. MARLBORO STANDARDIZATION:

A. **Objective:** Analytical and subjective evaluations of production Marlboro KS/LS.

B. **Status:** Subjective evaluations have been completed on the April 23, 1990 Marlboro pickup. A final report will be issued when analytical results are complete.

C. **Plans:** Issue final report on Marlboro April 23, 1990 pickup.

II. R.J. MARLBORO:

A. **Objective:** Development of a full-flavor product using all-natural flavor and blend components with Marlboro character.

B. **Status:** Based on subjective results, a new aftercut, new casing and new sheet material are being produced in large scale in Semiworks. These will be produced in both 16 mg and 13.5 mg to compare with current production.

C. **Plans:** Subjective evaluations are continuing.

III. INTERNATIONAL SUPPORT:

A. **Objective:** Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.

B. **Status:** Six (6) panels were completed during the reporting period.

PAN ASIAN MENTHOL - PROJECT CEDAR:

A. **Objective:** Development of free-standing menthol model to compete with Salem Lights (Hong Kong).

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B. **Status:** A factory trial was conducted and factory production was scheduled for early June.

CHESTERFIELD (JAPAN):

A. **Objective:** Develop a Chesterfield prototype for the Japanese market to compete with Lucky Strike.

B. **Status:** Additional flavor development of a Chesterfield prototype is in progress using new flavor blanks.

C. **Plans:** To investigate new flavor systems to make the product more similar to Lucky Strike.

DERBY MENTHOL (GUATEMALA):

A. **Objective:** Modification of Derby (Menthol) to compete with Casino (BAT) in the Guatemalan market.

B. **Status:** Prototypes have been received. Awaiting analyticals.

C. **Plans:** Subjective evaluations of the revised flavors will be conducted in Guatemala as well as in Richmond.

MERIT II (HONG KONG):

A. **Objective:** Develop a Merit prototype for the Hong Kong market to compete with Kent.

B. **Status:** Filler processed in large scale Semiworks production using Kent-type aftercut for PMI test vs. Kent in Hong Kong. Awaiting Kent cigarettes from Hong Kong and make pack of prototype.

NIPPON ART (JAPAN):

A. **Objective:** To develop an ART-type produced for the Japanese market.

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B. Status: Two flavor prototypes and an unflavored model have been made and will be tested on Danchi Panel with Caster Mild the first week of July pending analytical and subjective approval.

C. Plans: To evaluate finished Danchi "ART" cigarettes and modify flavors as required.

PROJECT CASTOR (JAPAN):

A. Objective: To develop Castor Mild product for Japan.

B. Status: Upon receipt of analytical results, the small scale models will be subjectively evaluated and flavor modifications will be made as needed.

C. Plans: To continue flavor development based on the subjective results from the small scale models.

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PROJECT NUMBER: 2307
PROJECT TITLE: Basic Flavor Investigation
WRITTEN BY: B. A. Demian
PROJECT LEADER: R. W. Hale
PERIOD COVERED: May, 1990

I. ANALYTICAL SUPPORT:

A. Objective: To provide analytical support for activities related to development and application of flavoring materials.

B. Status:

1. **Flavor Revisions:** GC and GC/MS purity checks and component identifications for 15 samples.

2. **Marlboro Family:** Thirty-one samples of Marlboro flavors, i.e., casings and aftercuts, and filler or cigarettes as well, were analyzed with a variety of techniques.

3. **Project Ambrosia:** Five different batches of "Aromatek 150" (ethylvanillin glucoside) were analyzed by UV-vis spectrophotometry and HPLC. The only difference among samples was a broad band absorption at wavelengths >360 nm., due to various amounts of a yellow impurity. Optical microscopy (135X) and a simple solubility test, as well as the aspect of the samples (free flowing powder versus wooly) indicates crystal structure polymorfism. Occurrence of polymorfism: two distinct polymorfs, was confirmed by powder X-ray diffraction pattern (analysis done by "Roche Labs"). It appears that the problems encountered at the formulation of "Aromatek 150" with EVA sideseam adhesive are related to this polymorfism. The less soluble polymorf can be formulated with the EVA adhesive without major changes in viscosity of the mixture.

   Four samples of coated cigarette paper or adhesive were analyzed by HPLC for flavor release compound content: one of the CR2910, CR2911 or "Aromatek 100".

4. **Natural Products:** Identification of honey extract components by GC/MS continues.

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5. **External Analytical Support:** Two samples of filter tow analyzed by Headspace/GC for residual solvents. Only acetone found (spinning solvent) in ration ~300:1 Control (FT107) versus Test (Diacel 3.0/35,000).

   Two samples of expanded tobacco compared by Likens-Nickerson steam distillation-extraction/HRGC/data analysis. Less distillable material found in the Test sample, as evidenced by ANOVA, Cluster analysis and Factor analysis of peak area data of the HRGC profiles, otherwide qualitatively identical.

II. BASIC FLAVOR INVESTIGATION:

A. **Objective:** To develop basic and applied knowledge for the purpose of flavor improvement or modification of existing products and to provide the basis for development and application of flavor technology for new, unique products.

B. **Status:**

1. **Low Tar/High Flavor:** Size Exclusion Chromatographic separation of Burley tobacco extract (batch J9AHR) continues.

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PROJECT NUMBER: 4009
PROJECT TITLE: Development Smoke Studies
PROJECT LEADER: B. L. Goodman
PERIOD COVERED: May, 1990

I. REDUCED SIDESTREAM CIGARETTES:

A. **Objective:** Develop subjectively acceptable cigarettes with reduced sidestream.

B. **Results:**

Trim: Superslim cigarettes were made in Louisville using papers coated with acids for subjective improvement. Cigarettes containing succinic acid on the outer wrap or inner liner did not show a subjective advantage over control Superslim cigarettes. Pimelic acid on the outer wrap gave a slight subjective improvement. There were no significant differences in analytical data or sidestream reduction for these cigarettes. Additional work on Superslims is planned with modified paper components.

Superslim cigarettes made with an outer wrap containing an equal mixture of Albacar and Multifex chalk were tested on the MC panel. No analytical differences were seen between this model and a control with only Multifex chalk (production paper) with the exception of burn rate, which was slower for the Albacar mixture. The panel did not rate the two cigarettes as being significantly different.

Lotus: The same paper with a mixture of Albacar and Multifex MM was also tested as a double wrap on a 24.8 mm circumference model. Again, the burn rate was slower for the mixed chalk by 0.6 puffs, with no other analytical or subjective differences. Sidestream visibility reduction averaged 66%, compared to 76% for the 17mm models.

The Sidestream Panel evaluated a number of cigarettes with a reduced level of mono-potassium phosphate combined with different acids on the high basis weight paper. Malonic acid with mono-potassium phosphate continues to be the leading candidate for subjective improvement. This paper is being evaluated with several new aftercuts.

C. **Plans:** Develop new papers for Superslims modifications and evaluate ash characteristics of paper modifications.

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Evaluate new aftercuts on full circumference models with phosphate-acid additives on the high basis weight paper.

II. SIDESTREAM VISIBILITY:

A. **Objective:** Determine sidestream visibility on experimental cigarettes, and test equipment for measuring visibility.

B. **Results:** A matrix test of variations in the level of malonic acid and monopotassium phosphate on the heavy basis weight paper showed decreases in sidestream with an increase in acid or phosphate. Acid levels up to 5% and phosphate up to 10% of the weight of the paper were used, with the subjectively preferred model having 7.5% phosphate and 3% acid. It gave 75% visibility reduction. The highest total additive level gave 84% reduction.

Medium (45 g/m^2) and high (63 g/m^2) basis weight low sidestream papers that had been coated with monopotassium phosphate for Project Ambrosia models were measured to give 51-61% visibility reduction for 24.8 mm king size cigarettes, and 56-68% reduction for the 24.0 circumference, 100 mm cigarettes. The heavy weight paper gave higher reductions in all cases than the medium weight paper. Some samples were also coated with 1% and 2% Aromatek-245. No clear trend was seen with the addition of Aromatek-245 flavor. Larger quantities of the same papers were coated at Kimberly-Clark for the POL tests.

Cigarette papers made at the University of Maine were also tested for visibility. Models with hydromagnesite and $Mg(OH)_2$ in a bilayer or as a composite sheet gave 64% and 74% reduction subjectively. Each paper had been coated with a 10% potassium succinate solution.

C. **Plans:** Measure visibility for current prototypes and conduct qualification tests with conventional cigarettes on the two eight port units.

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PROJECT NUMBER: 2304, 4015
PROJECT TITLE: Domestic Product Development
PROJECT LEADER: G. N. Yatrakis, J. B. Easley and J. L. Spruill
PERIOD COVERED: May, 1990

I. LOW TAR/HIGH FLAVOR

Project BOLD

A. **Objective:** Development of a 1mg 85mm and 2mg 100mm regular and menthol product competitive with Now and Carlton.

B. **Results:** Blend 240 was selected as an improvement over the initial blend used on POLs 0265, 0526 and 0530. Prototypes were made with this blend to evaluate new flavor systems. Based on evaluations, a model will be selected from this series for POL 0268.

C. **Plans:** Produce POL 0268 and evaluate a modified version of Blend 240 to lower puff count and increase subjective response.

II. PROJECT AMBROSIA

A. **Objective:** Develop a 23.0 circumference aromatic sidestream product as well as a 24.8 circumference 85mm and a 24.0 circumference 100mm and apply this technology to other products.

B. **Results:** Cigarette samples were made with Aromatek-100 (5%, 7%) and CR-2910 (5%, 7%) in the sideseam adhesive. Ambient and accelerated aged samples from these trials have been submitted for analysis of the flavor compound in the product.

Initials prototypes have been made to evaluate the R&D sideseam adhesive flow controller.

C. **Plans:** Complete evaluations of flow controller and aromatic sideseam prototypes.

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III. PROJECT AMBROSIA II

A. **Objective:** Develop K.S. and 100mm low smoke, low odor and low smoke/low odor prototypes.

B. **Results:** Thirty-one prototypes with various papers, filters and additives have been made and are being evaluated. Based on these evaluations, models will be chosen for POL testing in June. Several of these models are also undergoing accelerated aging tests.

C. **Plans:** Produce POL models. Evaluate accelerated aging tests.

IV. PROJECT MARLBORO

A. **Objective:** Design and develop a Marlboro 83mm FTB product at 13.5mg tar delivery and other Marlboro brand extensions.

B. **Results:** Products were designed using a 21mm filter and a 27mm filter at 13.5mg tar delivery as a Marlboro Medium between Full Flavor and Lights versions. POLs 0336 (83 w/21mm filter) and 0337 (83 w/27mm filter) have been produced and shipped to panelists for evaluation. Results due in late June.

Other Marlboro development work includes a 90mm product at 16mg tar and an 84mm product at 14mg tar.

C. **Plans:** Factory trial and Ad Pack scheduled for June utilizing the 83mm Medium design.

V. CAMBRIDGE IMPROVEMENT

A. **Objective:** To develop prototypes which will improve the cost effectiveness of the Cambridge brand family.

B. **Results:** Blend evaluation underway with POL 3636. Results available early June. Cost analysis of flax versus woodpulp cigarette paper and perforation versus non-perforation of tipping is under evaluation.

C. **Plans:** Pending POL results.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

VI. Brand Extensions & Modifications

A. **Objective:** Design and develop extensions and modifications to existing brand families.

B. **Results and Plans:**

Cambridge 100's U.L. @ 6mg tar - Design work completed.
Prototypes being produced.

Cambridge KS U.L. @ 6mg tar - Prototypes produced.
Undergoing analytical and subjective evaluation. Factory trial in June.

Cambridge 100's Menthol F.F. - Prototypes produced.
Awaiting analytical evaluations.

Marlboro 80 & 83mm Menthol FTB - Models produced and analyticals completed. Undergoing subjective evaluation. Factory trial in July.

Alpine 83mm Menthol - Design work completed. Models to be produced.

Bucks F.F. & Lights with A/C Modifications - Prototypes produced and analyticals completed. Subjective evaluations in progress to select a candidate for future POL evaluation.

Va. Slims KS with 20 & 23mm circumference - Design work completed for F.F., Lights, and U.L. models. Prototypes available in early June.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

PROJECT NUMBER: 4022
PROJECT TITLE: International Product Development
PROJECT LEADER: C. B. Altizer and R. E. Tinker
PERIOD COVERED: May, 1990

I. Philip Morris Super Lights Menthol (Hong Kong))

A. **Objective:** Develop a free-standing menthol product to compete favorably with Salem Lights at 8mg UK tar

B. **Results:** Topline results from PMI testing showed Salem Lights smokers significantly preferring their own brand versus Cedar II (60%/40%) and Cedar III (62%/38%).

C. **Plans:** Cedar III prototype to be launched as Philip Morris Super Lights Menthol July, 1990. Production start-up begins June 6th for an 18 million cigarette order.

II. Marlboro Monitor Programs (Several Countries)

A. **Objective:** Access acceptability of Marlboro in several Asian markets.

B. **Status:** Requests submitted for the following tests: Marlboro vs. Winston, Marlboro Lights vs. Kent (Hong Kong), 16mg Marlboro vs. 13.5mg Marlboro (Taiwan), locally produced Marlboro vs. USA Marlboro and locally produced Dunhill (Malaysia).

C. **Plans:** Ship all tests by October, 1990.

III. Merit Lights (Hong Kong)

A. **Objective:** Develop an 8-9mg UK tar product to favorably compete with Kent.

B. **Status:** Manufacturing PMI test of PM Super Lights blend/casing system with new aftercut versus Kent.

C. **Plans:** Ship PMI test week of June 25th.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

IV. Derby Lights Project (Guatemala)

A. **Objective:** Develop an acceptable line extended product to compete with BAT's Casino K.S. and Belmont E.S.

B. **Status:** Six Derby Lights prototypes have been received in Richmond for C.I. and subjective evaluations. Three samples incorporate the P2 Blend and three use the current Derby Blend with three AC systems recommended by Flavor Development.

C. **Plans:** Samples have been submitted for C.I.'s and subjective evaluations to determine which blend and flavor system is preferred.

V. Marlboro Improvement Project (Costa Rica)

A. **Objective:** Improve the Costa Rican Marlboro by modifying the tobacco blend and the use of U.S. flavor and casing concentrates.

B. **Status:** C.I.'s are complete and the Richmond Panel judged prototype 24B-1 acceptable. This prototype has U.S. casing and flavor concentrates and FTC Tar of 14.9 mg/cigt., 1.06 mg/cigt. nicotine.

C. **Plans:** Subjective and analytical results have been forwarded to Costa Rica for implementation.

VI. Project L&M K.S. (Guatemala)

A. **Objective:** Develop an acceptable L&M K.S. product for the Guatemalan market.

B. **Status:** C.I. results on L&M prototype Z6 from Guatemala delivered 16.3 mg/cigt. FTC Tar and .93 mg/cigt. nicotine.

C. **Plans:** Cigarette samples have been submitted to the Richmond Panel for subjective approval.

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VII. Marlboro Improvement Project (Venezuela)

A. **Objective:** Improve Marlboro by modifying tobacco blend, casings/flavors and cigarette construction to bring it closer to a U.S.A. Marlboro in deliveries and subjectives.

B. **Status:** Marlboro L.S. prototypes using U.S. casing/flavor concentrates and the current tobacco blend were subjectively evaluated in Venezuela vs. the current Marlboro by the Product Development team. Results indicate a preferences for the prototype with U.S. casings/flavors.

C. **Plans:** A consumer test will be conducted with current Marlboro and the Marlboro prototype with U.S. casing/flavors. A second step will be a blend change going to a U.S.A. Marlboro-type blend.

VIII. Project Winner (Venezuela)

A. **Objective:** Develop a local brand for Venezuela that is preferred by Belmont smokers.

B. **Status:** A Winner prototype using Fortuna tobacco blend and current Astor casings and flavors was preferred significantly by Belmont smokers on a consumer test vs. Belmont. The Product Development team also preferred the Winner prototype over Belmont.

C. **Plans:** The consumer test will be repeated (Winner V-1 vs. Belmont) to confirm results.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

PROJECT NUMBER: 5001
PROJECT TITLE: Packaging Studies
PROJECT LEADER: H. R. Dunaway
PERIOD COVERED: May, 1990

I. PACKAGING STUDIES

A. **Objective:** Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.

B. **Results:**

Residual Printing Solvents in Packaging Materials: Coordinated evaluation of the following items:

- Chesterfield Green Ink #89S403 (BASF)
- Replacement Additives for Phthalates in Inks (BASF)
- Marl 100 FT 20 (CHP - two samples comparing PPG & SCI water lacquers)
- Marl Lts KS FT 20 (CHP - two samples comparing PPG & SCI water lacquers)
- Saratoga 120 FT 20 (3F-950A/Alf)
- Gen Basic 100 FF SP 20 (L-641A/IPC)
- Gen Basic Lts 100 SP 20 (L-643A/IPC)
- Gen Basic Lts Men 100 SP 20 (L-645A/IPC)
- Gen Basic ULT 100 SP 20 (L-647A/IPC)
- PM Super Lts Men 62mm Tipping (11-155A/CHP)
- Printed Champagne Innerframe (Vitex)
- Next LT 100 SP 20 (4L-302D/Alf)
- PM# W-4350 Water Base Marl Red (DSI)
- Parliament Blue Ink #L-27157-C1 (American Ink)
- B&H Lts 100 SP 20 (4L-921A/Alf)
- B&H 100 Men SP 20 (3L-902A/Alf)
- L&M 80 FT 20/200/800 (51-802A/SP)
- PM Super Lts Men KS FT 20 (22-503A/SPW)
- PM Super Lts Men FT 20/200 (50-830A/SP)
- Next LT KS SP 20 (4L-300D/Alf)
- Ecusta 30036 Economy White Tipping
- Nicolmelt P-1662 Rewettable Adhesive (IGI)
- Henkel Foamaster B Defoamer

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Alkaline Process Label Stock: Packed samples of Marlboro KS labels were subjectively evaluated. Cigarettes packed in the alkaline process label were found to be comparable to those packed in the acid process control label.

Battistoni: Cigarettes packaged with domestically printed Battistoni blanks and cartons (Somerville Packaging) were subjectively evaluated and found to be comparable to those packed in the control Marlboro Lts materials.

Marlboro KS SP Labels: Based on subjective smoking results from two test matrices, developed with Purchasing Technical Services and Colonial Heights Packaging, a water base lacquer was recommended which allowed conversion to the new ink system at CHP. Testing continues to find acceptable alternative lacquers.

Gen Basic KS SP 20/200: A recommendation was made that cartons in this brand family not be used prior to two weeks after printing. Subsequent to unfavorable subjective smoking results in the initial evaluation of this offset printed carton, material sampled 15 days after printing was found to be acceptable.

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PROJECT NUMBER: 1307
PROJECT TITLE: Reconstituted Tobacco Development
PROJECT LEADER: R. G. Uhl
PERIOD COVERED: May, 1990

I. ART STEM UTILIZATION

A. **Objective:** Develop the capability to fully utilize spent absorber stems from the ART process.

B. **Results:** The status on qualifying the use of ART stems in reconstituted sheet feedstocks is as follows:

MPC Stems: POL 3637 containing RCB made with 2% MPC stems was mailed to panelists. The Scheduled POL closeout date is June 7. Completion of data analysis and issuing of final results is expected in early July.

CA Stems: Cigarettes to evaluate CA stems in sheet are being remade the week of 6/4. Two test models are being produced containing RCB made with 5% CA stems and RLTC made with 2% CA stems; one model contains RLB control sheet and the other contains RLB test sheet also made with 2% CA stems.

Bermuda Pilot Plant test runs were completed for the purpose of generating spent stem samples intended to differentiate the independent effects of citric acid casing, supercritical fluid, and filler extraction on the subjective quality of ART stems. The stem samples are undergoing rigorous analytical testing in an attempt to relate chemical and subjective changes. Pilot RL sheets incorporating 12% of each stem type were completed 5/22, and will be used for the subjective screening process.

C. **Plans:** Expedite internal panel testing of sheet materials made with CA stems to obtain a recommendation on POL testing. Complete subjective screening of pilot RL made with stems from various stages of ART processing.

II. CAST SHEET

A. **Objective:** Develop subjectively and physically acceptable reconstituted sheets for domestic and international application using cast sheet technology and proprietary binder systems.

B. **Results:** Flavor Development provided four flavor mixtures for use with guar gum cast sheets made with the Spanish tobacco blend. These have each been made into handsheets with and without the inclusion of 3% DAP as an added

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flavor ingredient. Handmade 100% cigarettes are submitted for subjective screening. Test sheets with and without 3% DAP, along with the respective "best" flavor system for that sheet, will be made at the TSA cast sheet facility in Cadiz, Spain when the facility is available for trials.

Evaluation of new binders continued. The potato starch recommended by KGF would not form a physically acceptable sheet even at high binder inclusion levels and was judged subjectively unacceptable (like RL baseweb with offnotes). The same performance is expected from other straight-chain starches. KGF is recommending several modified (branched) starches for evaluation.

Flour of the Konjac tuber (used in food preparation and approved for sheet testing) improves sheet physicals at low incorporation levels, although it does leave some "lumps" in the sheet. Subjective screening is pending.

Additional tamarind gums were evaluated, but none formed an acceptable sheet. A sample of the tamarind used by TSA in Spain (a 7/1 tamarind/guar blend) has been received and will be evaluated in handsheets.

TIC Corp. provided a sample of Colloid 3F (a mix of guar, pectin and alginate). This material is a low viscosity gum and gave acceptable sheet physical quality. Additional testing is pending subjective screening results.

A program was initiated to attempt the liberation of tobacco pectin by a combination of steam temperature/pressure treatment. This could allow the production of cast sheet without the need for chemical (DAP/ammonia) addition or added binders. The University of Maine TMP (thermomechanical pulping) unit, which does not encompass a rapid pressure release step, did not liberate the tobacco pectin. The feasibility of trials on a Sunds Defibrator TMP unit at Herty Foundation or on the batch digester/blow tank being installed at Maine is under evaluation.

C. Plans: Schedule cast sheet trials at the TSA facility in Cadiz, Spain using guar binders with and without DAP or added flavors; complete evaluation of the Spanish tamarind/guar binder to determine whether this should be included in the Cadiz test program.

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PROJECT NUMBER: 1308
PROJECT TITLE: Papermaking Process Development
PROJECT LEADER: R. M. Rogers
PERIOD COVERED: May 1990

I. LOW SIDESTREAM SMOKE PROJECT

A. **Objective:** Develop proprietary cigarette papers for low sidestream smoke.

B. **Results:** Standard low sidestream models have been completed on 18 of 21 samples from a matrix of magnesium compounds produced using the sol-gel process. These fillers typically exhibit slow drainage characteristics and produce extremely tight sheets. A few of the fillers can be successfully incorporated in cigarette papers and exhibit sidestream smoke reductions up to 70% although a portion of this performance can be attributed to extended static burn times. Additional sheets are being produced to evaluate filler production repeatability and the subjective impact of these materials.

A number of approaches are being pursued to identify the physical attributes of the fillers produced from the sol-gel process that inhibit the successful production of papers. The water holding capacity of the fillers appears to be a significant factor influencing drainage on a paper machine. This can be determined by evaluating the filler's response to solids separation with a centrifuge. Under standard treatment, fillers that exhibit poor drainage characteristics invariably can not be concentrated to high solids levels (>15%). The rheology characteristics of the fillers should also reflect their water holding capacity. Both approaches are being used to identify fillers with satisfactory physical characteristics and to direct work in developing fillers.

C. **Plans:** Develop a lab procedure to screen fillers for water retention characteristics and evaluate a select number of matrix samples (magnesium fillers from sol-gel process) in blends with calcium carbonate.

II. BANDED PAPERS (TOMORROW)

A. **Objective:** Incorporate cross directional bands of fiber and/or filler in cigarette papers in order to vary cigarette burn rate.

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B. Results: The Daubing Dandy has been modified to improve water release properties and band contrast (water release coating, direct drive and additional vacuum inserts). Laboratory work is being conducted to develop a fiber slurry with the correct rheology/tack properties to improve contrast and reduce the add-on weight. A number of commercial modified cellulose materials are being investigated. Two viable materials, micro-crystalline cellulose (chemical modification) and SOLKA-FLOC (mechanical modification) are both currently being evaluated in combination with a binder (guar gum or highly refined cellulose). Micro-fibrillated cellulose and Cellulon (experimental material produced using bacteria) are also being evaluated. The next set of trials are scheduled at Maine for the first week in June.

C. Plans: Complete laboratory evaluations of SOLKA-FLOC, micro-crystalline cellulose and micro-fibrillated cellulose.

Develop the capacity to apply bands with the Daubing Dandy at Maine (trials scheduled the first week in June).

III. PROPRIETARY FILTER MATERIAL

A. Objective: Develop a proprietary filter material in web form.

B. Results: Trials were completed at Maine this month using 1/8" cut CA staple (75% CA + 25% refined softwood). This material substantially improved paper machine runnability versus 1/4" CA staple. Dispersion of the CA and CA/cellulose by passing the mixtures through a refiner further enhanced machine runnability and improved visual formation. Porosity variance was equivalent to previous runs at Maine. The sheets were also sized with a number of binders (CMC or PVA) to enhance tensile and elongation properties. Numerous attempts to crepe the web at both the second press and the last dryer of the first dryer section were unsuccessful. Since creping the sheet is not a viable alternative at Maine, elongation properties of the sheet produced at Maine will remain below 2%. Pending evaluation of the current run of CA/cellulose web produced at Maine (physical and subjective), additional sheets will be produced at various porosities with and without binders.

The proprietary position of this web is dependent on developing a product with unique properties since the inclusion of <70% cellulose acetate cut staple in paper webs is prior art. It may be possible to alter the filtration characteristics of the web by substituting another form of cellulose (such as straw) for softwood.

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Incorporating crepe in the CA/cellulose web and/or reducing cut staple denier may also provide a proprietary position. A number of these options will be evaluated in the lab and handmade filter plugs constructed to determine directional characteristics. If evaluations are encouraging, additional sheets can be produced at Maine. If creping is required, agreements with a vendor can be initiated to develop pilot quantities or possibly production quantities of this material.

D. **Plans:** Produce and evaluate the physical properties of handsheets containing various levels of CA and types of cellulose fiber.

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PROJECT NUMBER: 1503
PROJECT TITLE: Modified Smoking Materials
PROJECT LEADER: W. A. Nichols
PERIOD COVERED: May, 1990

I. MOLTEN MENTHOL APPLICATION TO FOIL

A. **Objective:** Evaluate the use of a molten menthol applicator as a means of applying menthol to foil without the ethanol carrier.

B. **Results:** Previous experimentation has established the necessity for the applied menthol to be absorbed into the paper face of the pack foil. Testing is currently in progress to assess how absorption is effected by the sodium silicate adhesive used in laminating the paper and foil. If the adhesive is absorbed into the paper during lamination, absorption of molten menthol could be significantly inhibited

C. **Plans:** Alternate papers will be investigated to determine if the absorption rate of menthol into paper can be improved.

II. SIDESEAM ADHESIVE METERING SYSTEM

A. **Objective:** Evaluate a sideseam adhesive metering system, developed by Applied Technology, for use in the Semiworks.

B. **Results:** Working in collaboration with the Semiworks Process Control group, the sideseam adhesive system was packaged and installed in the Semiworks. Several modifications were made to the system to enhance the control of adhesive application. Currently, target application rates can be achieved with a variation of +/- 0.1 ml/min. The effects on control parameters due to interchange of glue nozzles are currently being studied. Significant improvement was achieved by using air pressure to close the nozzle versus spring pressure. Studies will be conducted to determine the accuracy of adhesive application with and without control and to determine the maximum amount of adhesive application without build-up on machine components.

C. **Plans:** Cigarette samples will be produced for Project Ambrosia. The process requirements for the production process will be defined.

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PROJECT NUMBER: 1806
PROJECT TITLE: New Tobacco Processes
PROJECT LEADER: T. C. Holland
PERIOD COVERED: May, 1990

I. MGC APPLICATION

A. **Objective:** Develop a process for applying an MGC menthol release compound to filler in the Primary to meet requirements for future new products.

B. **Results:** Work has been initiated to define equipment and process conditions required to provide the Semiworks with the capability to routinely apply MGC to support product development by July 1990.

Development Engineering has initiated a project to install a Witte separator for alcohol removal to support continuous Large scale processing of MGC application in the S/W. Current efforts are addressing the safety requirements of the alcohol removal equipment. Upon installation of the additional process equipment expected early July 1990, process trials will be conducted to establish process requirements for continuous application of MGC on filler.

Batch cylinder application studies have indicated that MGC can be successfully applied at a 3.0 wt % level with a 30/70 MGC/Ethanol solution. Success was based on minimum filler tackiness and short term (4000 cigarettes) Mark 8 maker trials. Larger quantities (200 pounds) of MGC applied filler were prepared to confirm findings with a longer run maker evaluation on the Mark 9 maker. If significant buildup on the maker is encountered, Central Engineering support will be requested to develop maker modifications to eliminate buildup.

Cigarettes produced from the batch cylinder will be evaluated to establish product stability with age and temperature. The studies will be conducted on cigarettes with both plain and charcoal filters.

MGC Application on the Maker

The #2 module, Mark 9 maker, at the S/W has been made available for the development of on the maker application of MGC. Work is underway to make the necessary equipment modifications required to begin the development testing. All modifications are expected to be completed by June 15, 1990. Support from Central Engineering will be required to support the maker development.

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C. **Plans:** Batch application trials have been scheduled to define the range of acceptable solutions and process conditions for successful MGC application.

II. PROJECT WARHOL

A. **Objective:** Provide support to FTR in conducting evaluation of European Marlboro ET expanded in the USA and US Marlboro ET expanded in Europe. Evaluation will identify the impact of tobaccos and process conditions on final Diet product quality.

B. **Status:** Support was provided to FTR in compiling additional process and quality information on the M/C segment of the Warhol trials. FTR has received all requested information from the M/C trials.

The TLA cigarettes from the Berlin trials have been made and submitted CI analysis.

C. **Plans:** Additional TLA samples will be obtained to provide analysis of firmness versus cigarette weight. All Warhol results will be completed and forwarded to FTR by the end of June 1990.

III. CIGARETTE QUALITY IMPROVEMENT FROM REDUCED "FINES" CUT FILLER

A. **Objective:** Establish the potential benefit in cigarette quality acquired by reducing tobacco fines (less than 1 mm particles) from the cut filler blend to demonstrate quality improvements claimed by GBE-Legg.

B. **Status:** The test plan to establish the benefits claimed by Legg has been revised.

C. **Plans:** The Rothman's Flow Thru Hopper will be obtained and installed on a maker to provide reduced breakage of the test filler. The testing will be initiated upon installation of the Flow Thru Hopper.

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PROJECT NUMBER: 1810
PROJECT TITLE: ART Process Development
PROJECT LEADER: R. Prasad
PERIOD COVERED: May, 1990

I. PROJECT ART - COMMERCIAL PLANT SUPPORT

A. **Objective:** To support the Bermuda Hundred Processing Facility startup/operation.

B. **Results:** This part of the program has been completed. The 12 Billion cigarettes/year capacity target is within reach with the cumulative effect of several changes in the cycle. The remaining items for commercial plant support are to transmit the backup distributor plate design to the plant personnel and to provide technical support during installation/startup of the CD monitor.

C. **Plans:** Complete the remaining items as per above.

II. PROJECT ART - BERMUDA HUNDRED PILOT PLANT

A. **Objective:** To support the commercial plant design and flavor development objectives at the Bermuda Hundred Pilot Plant.

B. **Results:**

Liquid Absorber Process - The commercial simulation trial of the countercurrent staging concept was completed with a total of 11 back-to-back extraction runs. Three stages with 20 gallons each of 6% CA/MPC solution (starting pH=3) were used for each run. As expected, the nicotine levels on each stage increased somewhat as the process lined out but had no adverse effect on absorption efficiency. The tobacco from these runs has been made into cigarettes by Flavor Development and is currently being smoked to determine the subjective impact of this mode of operation.

The pilot plant completed a series of runs designed to investigate the causes of batch-to-batch variations in extracted filler nicotine levels. A more extensive method was implemented for profiling the extracted filler bed for nicotine. The results show that the final nicotine level varied from about 0.06% at the bottom of the filler bed to about 0.15% at the top for standard liquid absorber process

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conditions (200 M/M). This variability within the tobacco bed has been responsible for much of the apparent batch-to-batch variation. This variation within the extracted filler bed occurs with stem as the absorbent as well, so it is strictly an extraction, rather than absorption, phenomena. Increasing the extraction time to 250 M/M reduces the variation to 0.06% - 0.09% throughout the filler bed. Additional extraction tests are currently in progress to fully characterize the nicotine distribution as a function of M/M.

C. **Plans:** Repeat the countercurrent staging series to confirm any flavor advantages.

III. PROJECT ART - NEW PROCESS DEVELOPMENT

A. **Objective:** To develop new processes for scale-up and implementation in the Bermuda Hundred Facility.

B. **Results:**

Liquid Absorber Nicotine Separation - Tests were conducted by Chemical Research on selective separation of nicotine from the liquid absorber effluent, with the objective of retaining flavor compounds while removing the nicotine. Reverse-phase separation fractions were generated using both C-18 silica gel and XAD-4 absorbents. The recovered neutral fractions were applied to extracted filler at two levels, representing 70 percent and 100 percent recovery. The resulting batches of filler were provided to Flavor Development for evaluation against control extracted product.

Liquid Absorber Effluent Disposal - Degradation of nicotine through conventional biological waste water treatment remains a promising method of disposal. The improved laboratory simulations of nicotine elimination through this method have been completed, and reviewed with Park 500 management. Further work will be conducted to evaluate biological pre-treatment of the liquid column effluent for nicotine destruction, prior to discharge into the waste water system.

C. **Plans:** Continue new process development via optimization of 1)absorber operation, 2)flavor separation, and 3)nicotine disposal steps.

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PROJECT NUMBER: 1811
PROJECT TITLE: Process Chemistry Development
PROJECT LEADER: S. E. Wrenn
PERIOD COVERED: May, 1990

I. MGC Studies

A. **Objective:** To determine the rheological and surface chemistry of MGC to support the application of this compound to tobacco filler or tobacco rod.

B. **Results:** Formulations of MGC with varying concentrations of ethanol, PG, and water are being made and tested for spray application onto filler. Physical characteristics (surface tension, rheology, contact angle, tackiness) are being quantified.

Stability of MGC with time, temperature and shear are being tested to determine the upper limits for processing conditions. Samples of MGC (77%) were held for one hour at 40, 50, and 60+C then submitted to Analytical Research for analysis of the isomer concentrations.

C. **Plans:** Determine the effect of time and temperature on the concentration of free menthol collected on cellulose acetate filters which are equilibrated with filler cased with MGC.

II. CAST SHEET DEVELOPMENT

A. **Objective:** To investigate alternate binder systems to be used as an alternative to the current sheet processes.

B. **Results:** Several gums were incorporated into a recon sheet in an attempt to produce a sheet material from a blend of tobacco dust and washed burley stem with good physical properties which could be used as a replacement for a blend of RCB and the RL's. Potato starch was suggested by General Foods (H. Stahl). A sheet product using 14% potato starch was made and subjectively evaluated by J. Swain. He concluded that this starch produced a 'baseweb, with off-notes' subjective response. His recommendation was to discontinue efforts with this binder. Based on the subjective response and the quantity of starch which was required to produce a low quality sheet, no additional work will be done with this binder. However, Dr. Stahl is compiling a list of modified starches which may contribute specific characteristics to a sheet product. These suggested binders will be evaluated after review of the physical and chemical characteristics.

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Konjac flour, a product produced by FMC, was suggested by Dr. Stahl because of the film formation of the binder. A small percent addition of this material may be used to enhance sheet performance of a lesser binder. Konjac reacts with addition of bases to form a stronger film. This material has been received and approved for smoking. Evaluations of Konjac are being made. Sheets with Konjac as the only binder will be produced for a subjective evaluation.

* Samples of Tamarind gum were received from TIC. The films which were produced are similar to starch film and do not appear to have carrying power. To date no continuous sheet has been prepared form Tamarind gum.

A sample of a gum blend was received form TIC which was a 'best guess' on their part as to the binder which would make a good tobacco sheet. The blend of gums, Colloid 3F, is a mixture of alginate, pectin and guar. Samples of recon sheet were prepared with a 9.1% addition of the gum for subjective and physicals. The gum solution is a low viscosity which may allow for sheet production at a high (<20%) solids content. After preliminary subjective screening, sheet formulation will continue to optimize physical characteristics.

A mechanical casting unit was obtained form Gardener Instruments to investigate the parameters of casting. Guar sheets were prepared using the caster varying speed of the knife pull and submitted for physical measurements. Additionally, a test plan is being written to determine the variation from batch to batch and from sheet 1 to sheet 4 within a batch.

C. **Plans:** Continue investigations of water soluble gums. Investigate modified starches and tapioca starch sheet binders.

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PROJECT NUMBER: 1812
PROJECT TITLE: New Expanded Tobacco
PROJECT LEADER: W. D. Winterson
PERIOD COVERED: May, 1990

I. NEW EXPANSION PROCESS DEVELOPMENT

A. **Objective:** Develop tobacco expansion technology and associated processing for the production of expanded tobacco materials having the physical and subjective characteristics required to support future product needs.

B. **Results:** Carbon dioxide gas impregnation studies continued. A test grid is being run over a range of impregnation temperatures and pressures to quantify the effects of these variables on gas pick up and retention. Temperature has a critical impact for a given pressure. Adequate pick up has been observed at pressures as low as 300 psig. A test was run at General Foods which demonstrated the twin screw extruder was not capable of holding adequate impregnation pressure in a CO2 - tobacco contact zone. An initial test using SF6 as an impregnant was run at low pressure with little expansion observed, it will be retested at a higher pressure.

Attempts to use the thermal treatment unit as a constant moisture thermal setting device were unsuccessful due to excessive steam condensation. An alternative bench top design using superheated steam on flowing tobacco was developed with Development Engineering. Discussions are continuing with high temperature, short residence time flash dryer manufacturers including Bepex, CE Raymond, and Jet-O-Drier to arrange and execute equipment trials. Tentative dates have been established in June and July for tests.

Objectives and plans are being developed to evaluate whole blend partial expansion, both using the present DIET process and through alternate processes. Work is proceeding on a program to model the tower and separator with CHAM, a process modeling firm.

C. **Plans:** Continue gas phase impregnation studies, investigate the fundamental mechanisms of tobacco expansion and collapse, and pursue methods of continuous gas phase impregnation. Test thermal setting of tobacco while minimizing changes in tobacco moisture content. Evaluate alternate expansion techniques. Initiate program on partial expansion and on computer modeling of the tower and separator.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

II. DIET PROCESS IMPROVEMENTS

A. **Objective:** Develop improvements to the existing DIET process for use if expansion production capacity is increased prior to completion of the new process development effort.

B. **Results:** The first round of testing was completed on the plexiglass model of the tower and separator at PEMM-CORP. Plug flow was achieved using a baffle in the tangential separator. A drop test was run to evaluate the effect of drop distance on breakage of frozen impregnated filler.

C. **Plans:** Prepare plans and implement to develop design information for inclusion in the planned DIET line addition at Cabarrus.

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PROJECT NUMBER: 1101
PROJECT TITLE: Entomological Research
PROJECT LEADER: D. L. Faustini
WRITTEN BY: L. Ryan
PERIOD COVERED: May, 1990

I. METHOPRENE

A. **Objective:** Assist with implementation of methoprene into PM's cigarette beetle (CB) management program.

B. **Results:** Collaborative studies were completed with Engineering and RLPF personnel on the of Kabat® application to whole leaf in the pre-threshing ordering cylinder. This application achieves uniform treatment of lamina, but does not adequately treat stems (1). Final test results of cylinder application of Kabat® to ET were reported (2) and the first trial was run to study the fate of methoprene through the DIET process. Official management approval to treat 100% of the 1990 flue-cured lamina with 5 ppm methoprene was communicated to PM USA personnel (3).

C. **Plans:** Integrate methoprene into PM's pest management program. Cease work on cylinder applications of Kabat®. Investigate efficacy of methoprene against feral CBs compared to lab colonies (4,5) and compare the efficacy of S- to R,S-methoprene (6).

D. **References:**

1. Sweeney, W. R. Kabat® on Total Leaf Test Results. Memo to Richardson, E. E. 4 April 1990.

2. Ryan, L. Results of Kabat® Cylinder Spraying onto ET. Notebook No. 8533, pp. 121, 123 and 124. Communicated to Sweeney, W. R. 10 April 1990.

3. Charles, J. L. 100% Kabat®-treated Flue-Cured Lamina. Presentation to PM USA Review Meeting: Louisville. 8 May 1990.

4. Lehman, R. M. Notebook No. 8740, pp. 85-86.

5. Minor, M. F. Notebook No. 8740, p. 84.

6. Lehman, R. M. Notebook No. 8740, pp. 87-89.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

II. SERVICE TO OTHERS

A. Objective: Provide technical services to areas outside R&D.

B. Results: Test insects (***Ephestia kuehniella*** and ***Stegobium paniceum***) were supplied to Miller Brewing Co. for a CO_2 fumigation evaluation (1). Advice was given to PM Europe on assessing potentially acceptable pesticides for use on PM property (2). Cigarette beetles and colony maintenance advice were given to QA personnel at Cabarrus (3).

C. Plans: Provide technical assistance as requested. Advise PM Europe on fumigation procedures to impact leaf supplies (4). Initiate studies aimed at reducing the duration of non-probe vacuum-steam conditioning cycles at 20th Street while qualifying for phytosanitary certification (5).

D. References:

1. Faustini, D. L. Test Insects for CO_2 Fumigation. Letter to Thompson, R. of Miller Brewing Co. 3 May 1990.

2. Faustini, D. L. PM Approved Pesticides. Memo to Freymond, J.-M. 9 April 1990.

3. Minor, M. F. Telephone conversation with Jerome, A. M. of Cabarrus, QA. 18 April 1990.

4. Schulthess, D. PME Fumigation Policy. Memo to Distribution. 30 April 1990.

5. Faustini, D. L. Phytosanitary Certification of Export Blended Leaf. Memo to Distribution. 3 May 1990.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

PROJECT NUMBER: 1620
PROJECT TITLE: Electrophysiological Studies
PROJECT LEADER: F. P. Gullotta
WRITTEN BY: C. S. Hayes
PERIOD COVERED: May, 1990

I. PATTERN-REVERSAL EVOKED POTENTIALS (PREPs)

A. **Objective:** To objectively and reliably evaluate human responses to cigarettes.

B. **Results:**

1. PREP - Impact Study

Work on this study continues. The objective of the study is to compare the electrophysiological and subjective effects of ART-blend cigarettes made from varying proportions of extracted and unextracted fillers. To date, the study is 80% completed.[1]

2. PREP - Nicotine/Menthol Interaction Study

Previous work comparing cigarettes made from extracted and unextracted fillers has shown that menthol increases impact in the former and decreases impact in the latter. Most importantly, a statistically significant association between impact scores and PREP P_1 - N_2 amplitudes was demonstrated. Specifically, higher impact scores were associated with larger increases in P_1 - N_2 amplitudes.[2] In order to further investigate menthol/nicotine interactions on perceived impact, a PREP study has been initiated to determine whether objective correlates of anticipated nicotine/menthol interactions can be obtained.

The PREP study is employing factorial combinations of four levels of menthol (0.00, 0.04, 0.08 and 0.16 mg/puff) and three levels of nicotine (0.08, 0.51 and 1.10 mg/puff) on ART cigarettes. Thus, twelve experimental cigarettes with various combinations of menthol and nicotine are being tested. PREPs are being recorded in twenty R&D smokers before and after the smoking of each of the twelve cigarettes. Additionally, at the end of each experimental session, subjects are required to complete a ballot evaluating the cigarette that was smoked. To date, testing has been initiated and should be completed by early September.

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C. **Plans:** Complete the testing of twenty subjects with each of six ART models from the impact study and analyze the data. Continue testing of twenty subjects with each of twelve ART models from the nicotine/interaction study.

D. **References:**

1. Martin, B. R. Notebook No. 8931, pp. 115-159.

2. Gullotta, F. P., Hayes, C. S. and Martin, B. R. Phase I ART Study. Memo to H. L. Spielberg; 1990 March 29.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

PROJECT NUMBER: 1702
PROJECT TITLE: Optical Processing and Aerosol Research
PROJECT LEADER: K. A. Cox
PERIOD COVERED: May, 1990

I. PACK INSPECTION SYSTEM

A. **Objective:** Develop and implement an on-line cigarette pack inspection system.

B. **Results:** An advisory group, chaired by Miles Waugh, was assembled to review the design of OSIRIS, the factory prototype pack inspection system. The advisory group members represent Manufacturing, Quality Engineering, Engineering Applied Technology, Engineering Manufacturing and Quality Audit. A document describing the operational concepts of the pack inspection system was completed and distributed to the members of the advisory group. The group was later convened and a description of the features and operation of OSIRIS provided. The comments from the group members were very valuable. Modifications have been made to the software design to incorporate a number of the suggestions. The group will continue to meet periodically throughout the development period.

A search is underway to identify a business partner to serve as a vendor for OSIRIS. The vendor will be expected to manufacture, support and sustain an ongoing development effort to maintain OSIRIS at the state-of-the-art. A meeting was held with representatives from Modicon, Inc., to discuss the possibility of their assuming this role. Similar meetings are being scheduled with additional candidates.

C. **Plans:** Identify a vendor for OSIRIS. Complete the software design and begin coding.

E. **References:** Miles Waugh, OSIRIS Advisory Group Meeting, memo to R. Davidson, C. Irving and E. Richardson, May 14, 1990.

II. PACKAGE BLANK INSPECTION SYSTEM

A. **Objective:** Develop an off-line inspection system for the high resolution comparison of package blanks with the corresponding master artwork.

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B. **Results:** The package blank inspection system was installed in the QA Department in the middle of March. The system has been performing well but has required more time per inspection than is desired. During this past month, the software was modified to decrease the inspection time from 3 minutes and 45 seconds to 2 minutes and 45 seconds.

C. **Plans:** Identify commercial imaging systems supported on a Micro Vax or a PC that can be used to replace the current Sun-Androx implementation.

III. PRINT WEB INSPECTION

A. **Objective:** Develop a system for the global inspection of print web on the printing press.

B. **Results:** A simple algorithm was proposed for the global inspection of print web. The algorithm differs from that currently used for the inspection of cigarette packs. It takes advantage of the reduced image distortion and provides the needed capability for the detection of much smaller defects.

A meeting was held with representatives from Mercury Computer Systems, Inc., (MCS) to discuss implementation of the inspection using their hardware. The MCS hardware was found to be poorly suited to the application. We are continuing to evaluate alternative solutions based upon commercial hardware. Keeping the system affordable is presently a major concern.

C. **Plans:** Complete the survey of currently available technology for web inspection and formulate a development plan.

IV. AEROSOL RESEARCH

A. **Objective:** Generalize the technique for measuring the size distribution of aerosols to take into account absorption.

B. **Results:** Our current method for the determination of the particle size distribution of the cigarette smoke aerosol is based upon spectral extinction measurements. In using this method, it is assumed that the absorption of the aerosol particles is negligible and can be neglected relative to the scattering contribution to extinction. This assumption was necessary due to our lack of knowledge of the

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absorption characteristics of tobacco smoke over the wavelength range from 400-900 nanometers. We have recently carried out accurate measurements of the absorption coefficient of tobacco smoke over this wavelength range. During this past month, the method for obtaining the particle size distribution from the spectral extinction measurements has been generalized to take into account the absorption. The effect has been found to be small for cigarette smoke particles at wavelengths above 450 nanometers. However, taking into account absorption will be important in allowing us to consider shorter wavelengths and smaller particles.

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PROJECT NUMBER: 1704
PROJECT TITLE: Supercritical Fluid Processes
PROJECT LEADER: T. M. Howell
PERIOD COVERED: May, 1990

I. EXPANDED TOBACCO

A. **Objective:** Support new DIET process development.

B. **Results:** The solubility of CO2 in water containing tobacco solubles was compared to the solubility of CO2 in pure water at 25 °C. and at pressures up to 800 psig. Six aliquots of uncased bright tobacco, JOQG, were washed with 1000 cc. of HPLC grade water in a counter-current fashion in order to obtain the water saturated with tobacco solubles. The measured solubility values for CO2 in pure water agreed within 4.0% of literature data.

C. **Conclusions:** The presence of tobacco solubles inhibits the solubility of carbon dioxide in water. In the above mentioned solution, CO2 solubility in water was reduced by about 30%.

D. **Plans:** To repeat the measurement on a separate solution saturated with tobacco solubles.

E. **References:** Weibe, R., Gaddy, V. L., J. Am. Chem. Soc., 62, 815-7, 1940.

II. EXPANDED TOBACCO

A. **Objective:** Support new DIET process development.

B. **Results:** An experimental test grid was developed and initiated to investigate the effect of pressure, temperature, and OV on CO2 pickup by tobacco. The test will also include determining the stability of the retained CO2 at atmospheric conditions. The criterion for stability will be based on the total amount of CO2 remaining after the tobacco stands for 30 minutes at room conditions. This stability is important for determining the proximity of the expansion tower to the impregnator.

D. **Plans:** Complete the grid study.

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PROJECT NUMBER: 1708
PROJECT TITLE: Physical Chemistry and Process Monitoring
PROJECT LEADER: J. L. Banyasz
PERIOD COVERED: May, 1990

I. NEW EXPANSION PROCESSES (J. Crump and S. Ganeriwala)

A. **Objective:** Evaluate and quantify the variables affecting the collapse of expanded tobacco.

B. **Results:** Stress relaxation and recovery tests were carried out with bright lamina to simulate expansion and collapse using the Dynastat. The lamina were conditioned to 75% RH (16% OV) and then stretched by 50-100% using the "zero span" technique. The strain was then maintained constant and the time dependence of the stress was monitored. The stress relaxation rates were found to be very rapid with the stress falling to 33% of its initial value within one second. The relaxation rate did not appear to be a strong function of the initial strain. Experiments were also conducted where the sample was initially stretched and then allowed to recover. The recovery rates were about 50% slower than the stress relaxation rates. The results indicate that tobacco would expand readily and collapse rapidly at 16% OV. Further experiments will be carried out as a function of OV and tobacco stiffness.

Experiments have been initiated to determine the feasibility of using the FTMA as a means of monitoring the collapse of expanded filler in real time.

Tower exit expanded tobacco was reordered to OV values ranging from 5 to 35%. Both the reordering rates and the final OV values varied during the process. The resulting SV values were found to decrease in an approximately sigmoidal fashion as the final OV increased. The steepest portion of the curve appears to be around 60% RH (11.5 to 12% OV). The results indicate the collapse is sensitive to both the reordering rate and the final OV value. Experiments are continuing to determine SV as a function of final OV at constant rates of reordering.

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II. PROJECT ART (NICOTINE STUDIES) (A. K. Basak)

A. Objective: Characterize the behavior of nicotine in aqueous solution.

B. Results: Titrations are in progress to determine the interactions between nicotine, citrate, and carbon dioxide in concentrated solutions.

III. MENTHOL STUDIES (T. V. Van Auken)

A. Objective: Determine the mechanism of menthol migration.

B. Results: Modifications to the tow mentholator have been completed. Testing will commence next week.

IV. OPERATIONS SUPPORT (D. M. Driscoll)

A. Objective: Develop and implement at plant levels, low energy and low capital cost technology to assure the required levels of dissolved oxygen (4.6 mg/L) in the effluent from the Park 500 waste-treatment plant.

B. Results: The oxygenation process using the sonic mixer has been scaled to 50 gpm level. The results suggest that efficiency increases with flow rate probably due to increased turbulence. Scale up will continue pending the arrival of a larger mixer.

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PROJECT NUMBER: 1720
PROJECT TITLE: Analytical Microscopy
PROJECT LEADER: V. L. Baliga
PERIOD COVERED: May, 1990

I. REDUCED SIDESTREAM/FILTRATION/PAPER TECHNOLOGY (SANDERS, YI)

A. **Objective:** Examine the ultrastructure of selected cigarette papers, paper additives, and filtration materials in support of the paper technology project.

B. **Results:**

Chemical Powders: Twenty five magnesium-containing products that were prepared from magnesium methyl carbonate ($MgMeCO_3$) with the amounts of $MgMeCO_3$ and KOH varied and the reaction temperatures and reaction times varied were examined for structural and elemental differences. Five major particle types were identified from the samples. They included an acicular rod-shaped structure that resembled nesquehonite ($MgCO_3 \cdot 3H_2O$), a spherical to ovoid cluster of platelets that resembled platelets of hydromagnesite ($Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$), flattened platelets that resembled $Mg(OH)_2$, equant particles in loosely packed clusters, and an 'amorphous' structure with little to no crystalline shape.[1,2,3,4,5]

Papers: Two cigarette papers with different sources of Multifex MM $CaCO_3$ were examined to determine the structural differences of the two forms of $CaCO_3$. The paper made with domestic $CaCO_3$ from Pfizer consisted of large aggregates that measured about 10μm in diameter while the imported $CaCO_3$, used in Superslims, was evenly dispersed throughout the paper and had fewer and smaller aggregates.[6]

C. **Conclusions:** The products synthesized from $MgMeCO_3$ that exhibited an amorphous structure tended to reduce sidestream smoke more than the products that were more crystalline in form.

D. **Plans:** Papers that were made with four crystalline types of $MgMeCO_3$ components - the acicular rod, the spherical platelet, the flat platelet, and the amorphous structure - will be examined to determine if the crystal shape found in the paper is similar to the dried powder, starting material.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

E. **References:**

1. Sanders, K., "Examination of Three Mg Containing Particles," Memo to K. Podraza, April 17, 1990.
2. Sanders, K., Examination of Four Mg Containing Particles," Memo to K. Podraza, April 20, 1990.
3. Sanders, K., "Examination of Five Mg Containing Particles," Memo to K. Podraza, April 25, 1990.
4. Sanders, K., "Examination of Three Mg Containing Particles," Memo to K. Podraza, May 7, 1990.
5. Yi, G., "Various Forms of Dried Products from Magnesium Methyl Carbonate Reactants," Memo to K. Podraza, May 7, 1990.
6. Yi, G., "Comparisons of MM CaCO, Particles from Superslims and Pfizer Cigarette Papers," May, 17, 1990.

II. OPERATIONS SUPPORT (BALIGA)

A. **Objective:** Provide analytical support to operations.

B. **Results:**

Two samples of coated, menthol encapsulated pellets and one sample of coated, menthol encapsulated string were examined to determine the uniformity of the coating material. The coating material uniformily surrounded one set of pellets and the string while the second set of pellets was partially coated. The honeycomb interior of the string contained unit cells that were two to five times smaller than those in the interior of the pellets[1] .

One washer gasket was examined and was found to contain Si and Mg and was fibrous in structure[2] .

C. **References:**

1. Baliga, V., "Structural and Elemental Analysis of Coated, Encapsulated Menthol Pellets and Coated, Encapsulated Menthol String," Memo to J. Washington, April 27, 1990.
2. Baliga, V., P.M. Notebook #8412, p. 31.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

PROJECT NUMBER: 1752
PROJECT TITLE: Molecular Structure Determination
PROJECT LEADER: G. Vilcins
PERIOD COVERED: May, 1990

I. NMR ANALYSIS OF CIGARETTE PAPER

A. Objective: To determine the non-cellulose components in modified cigarette papers.

B. Results: Paper samples treated with various acetylating agents under different conditions were examined by NMR to determine the degree of acetate substitution (DOS), the number of acetate groups per cellulose glycosyl unit.

C. Conclusions: DOS values ranged from 0 to 0.3 depending on the acetylation procedure applied. It was demonstrated that the NMR procedure is an effective method for the quantitative determination of intact paper.

D. References:

Wooten, J., "^{13}C CPMAS NMR Spectra of Acetylated Paper - III," memo to D. Simpson, May 2, 1990.

II. ART STEMS CHARACTERIZATION

A. Objective: The identification of major chemical changes in stems resulting from exposure to supercritical carbon dioxide, supercritical carbon dioxide solubles (SCCS) of filler, and the addition of citric acid.

B. Results:

A sampling matrix was structured so that chemical changes to the stems could be traced to the general areas of the ART process. Three extractions were performed at the Bermuda Hundred Pilot Plant with citric acid treated stems and no filler, citric acid treated stems and filler, and citric acid untreated stems, and no filler, respectively. Each extraction was performed at 2000 psi and 140° F for 2.5 hrs. Stem samples were collected before and after each extraction. Bomb samples of the supercritical carbon dioxide also were obtained at the end of each extraction and from the working vessel prior to the second extraction.

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The weight percent of SCCS was determined for each hexane wash. The hexane solutions were analyzed by gas chromatography, gas chromatography/mass spectrometry, and nuclear magnetic resonance spectrometry. Stem samples have been submitted for analysis by microscopy, supercritical fluid extraction/gas chromatography, evolved gas analysis/Fourier transform infrared spectrometry, thermal chromatography/mass spectrometry, and fast atom bombardment mass spectrometry.

C. **Plans:** Work will be continued.

D. **References:**

1. Shafer, K., PM Notebook #8789, p. 70.
2. Jensen,N., "Mass Spectral Analysis of Bomb Samples from Stems Studies," memo to K. Shafer, May 16, 1990.
3. Bassfield, R., "Monthly Report, May 1990," memo to G. Vilcins, May 22, 1990.
4. Lyons-Hart, J., PM Notebook #7820, p. 61.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

PROJECT NUMBER: 1757
PROJECT TITLE: Analytical Flavor Specifications
PROJECT LEADER: M. L. Zimmermann
PERIOD COVERED: May, 1990

FLAVOR SPECIFICATIONS AND CERTIFICATION

A. **Objective:** To develop analytical and sensory specifications for current, incoming flavor materials and to transmit specifications and methods for monitoring specifications to appropriate groups. To certify that PMI export flavor materials meet GFO, to issue a certification of analysis and to transfer methodology and certification to the Flavor Center.

B. **Results:**

German certification continues for the PMI export materials not covered by the first round of testing. A certification of analysis was issued for each of the materials used in both the export and domestic formulations.

Specifications were completed for 95 of the direct material codes and turned over to the various groups involved. Data transfer from our system to the IBM system 36 is now being evaluated. This completes materials for the first three large vendors for a total vendor count of seventeen out of sixty. The dry flavors, FRG Direct Materials, and the fourth vendor are currently being analyzed. The materials for the fifth vendor have been requested. An update meeting was held to disseminate this information in detail.

South American flavor materials were examined for marker/contaminant content. Vendor preshipments, CR-samples, and Flavor Center materials were also analyzed for contaminant content.

Fourteen samples were analyzed by HPLC for specification development. Routine analyses for flavor fingerprint, sugars and xanthine determinations were conducted. Five additional samples were tested by HPLC for compliance to German regulations. Sensory evaluation was also conducted for specification purposes. Routine HPLC maintenance was conducted during this period.

C. **Plans:** Continue the certification of the PMI samples, begin the specifications for Dry Flavors, FRG Direct Materials, and the fourth vendor.

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PROJECT NUMBER: 1759
PROJECT TITLE: Materials Evaluation and Elemental Analysis
PROJECT LEADER: P. F. Grantham
PERIOD COVERED: May, 1990

I. MATERIALS EVALUATION

A. **Objective:** To identify components of commercial products prior to their use at PM facilities.

B. **Results:** Routine analysis of commercial products, Packaging Development, and TTG samples continue. The Materials Evaluation Committee conducted audits at MC Primary and at 20th Street. Several foreign matter and customer complaint samples were analyzed this month.

C. **Plans:** Analyses of samples are on-going. Presentations will be made upon request. Audits will be made as scheduled.

II. ELEMENTAL ANALYSIS

A. **Objective:** To provide qualitative and quantitative elemental data on tobacco, cigarette paper, development and materials evaluation samples.

B. **Results:**

The X-ray Fluorescence (XRF) method for screening selected elements in packaging material has been used to screen approximately twenty samples associated with the Marlboro Brand. Samples will continue to be analyzed as they are received from Incoming Materials QA.

A potentiomentric titration assay was used to determine % Menthylchloroformate in three production samples of menthol release compounds.

Samples of Vanil-3 and Glucosementhylcarbonate (GMC) were analyzed using an ion-selective electrode to determine if fluoride was present. No fluoride was detected in either sample. The GMC sample was also analyzed to determine chloride concentration using an ion-selective electrode. Duplicate runs of the sample yielded approximately 700µg/g chloride.

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Differential Scanning Calorimetry (DSC) was used to determine the specific heat capacity of a control PVA adhesive and a PVA adhesive containing 5% CR-2910, a flavor release compound. The control sample showed a gradual increase in specific heat capacity between 23°C and 55°C. This is most likely due to the contribution of the heat of vaporization of low boiling volatiles present in the adhesive. The sample containing 5% CR-2910 showed very little variation over the temperature range.

Thermogravimetric/Differential Thermal Analysis (TG/DTA) was used to analyze samples of hydromagnesite, hydrotalcite, magnesium hydroxide, and potassium succinate. All samples were heated from ambient temperature to 1000°C at rates of 10° or 20° per minute. The samples of hydrotalcite showed multiple endothermic transitions over the temperature range. The magnesium hydroxide samples varied. One sample showed a single endothermic transition at 330°C, another had a single transition at 378°C and a third showed multiple transitions at 327°C and 367°C. The single potassium succinate sample gave endothermic transitions at 58°C, 252°C and 891°C. Thermogravimetry showed total weight loss for the potassium succinate, indicating complete volatilization of the sample.

C. Plans: Support for R&D projects for elemental analysis will continue.

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PROJECT NUMBER: 1902
PROJECT TITLE: Cell/Tissue Culture Research
PROJECT LEADER: D. Teng
WRITTEN BY: M. Shulleeta
PERIOD COVERED: May, 1990

I. TOBACCO MICROBIOLOGY

A. Objective: To develop methods and to evaluate the microflora resident in tobacco materials.

B. Results:

1. Bright Casing Storage Study

Samples of bright casing stored at 4, 27, 37.5 and 50°C have been analyzed for changes in microbial populations after 0, 24 hours, 48 hours, 1 week and 2 weeks of storage. To date, no detectable microbial population has been observed in any of the analyzed samples (1).

2. Enumeration of Bacteria

In order to investigate alternative methods for the reliable determination of bacterial numbers associated with tobacco, the Most Probable Number (MPN) method via the use of the Vitek Bioburden Enumeration Card was compared to our standard operating procedure (SOP) for viable plate counts (2). The bacterial counts from DBC burley tobacco obtained with the two methods were not comparable in 3 experiments with 3 replicates per experiment (3).

3. Determination of Ergosterol

The FTR procedure for the determination of ergosterol (4) was evaluated as an alternative indicator of mold growth in tobacco. The HPLC standard curve for ergosterol produced excellent linearity ($R^2 > 0.99$) in the 1 - 50 µg/ml range (5). A peak from DBC bright tobacco with an elution time of 46.5 minutes could not be eliminated with the silica gel cleanup recommended. Its presence lengthens the HPLC time considerably since ergosterol elutes at 8.6 minutes.

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C. **Plans:**

1. Continue the Bright Casing storage study and perform analyses after 1, 2, 3 and 4 months.

2. Issue a memo reporting the findings regarding the enumeration of bacteria via MPN.

3. Attempt to eliminate the 46.5 minute peak present in HPLC determinations of ergosterol.

D. **References:**

1. Chadick, D. Notebook No. 8825, p. 156.

2. Crockett, E. Special Report Number 86-055, 1986 February 20.

3. Gaines, O. Notebook No. 8904, pp. 67-70, 76-98.

4. Bindler, G. N., Piadé, J. J. and Schulthess, D. Evaluation of selected steroids as chemical markers of past or presently occurring fungal infections on tobacco. *Beitrage zur tabakforschung*. 14(2): 127-134; 1988 October.

5. Weissbecker, L. Notebook No. 8822, p. 127.

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Source: https://www.industrydocuments.ucsf.edu/docs/rrxm0000

PROJECT NUMBER: 1904
PROJECT TITLE: Tobacco Physiology and Biochemistry
PROJECT LEADER: D. J. Ayers
WRITTEN BY: H. Y. Nakatani
PERIOD COVERED: May, 1990

I. LOW NICOTINE STUDY

A. **Objective:** To investigate the biochemistry of the nicotine biosynthetic pathway at the putrescine N-methyltransferase (PMT) level and specifically to isolate PMT from tobacco root extracts.

B. **Results:** The phenyl-Sepharose samples from Group 22 have been processed through the DEAE/ω-amino-Sepharose (affinity-mode) columns to generate Batch 3 (2,3). All of the roots from the Group 23 hydroponically grown Burley 21 tobacco plants have been harvested and stored at -80° C (1,2). A number of samples have been further processed through the phenyl-Sepharose column (1).

The protein levels of various PMT samples determined by the Kendrick Laboratory (KL) and PM methods were compared. For samples with low protein concentrations, the KL/PM ratios ranged from 0.63 to 0.68 whereas for samples with high protein concentrations, the ratio varied more widely between 0.32 to 0.57 (3).

The proteins in the PMT-active regions have been extracted from the agarose IEF (isoelectric focusing) gels for the Batch 1 samples and are being examined by SDS-PAGE. Carbonic anhydrase (Enprotech) appears to be a satisfactory protein standard for comparison purposes of protein levels (4).

A pooled sample from Batch 2 was fractionated by HPLC gel permeation columns for the further purification of the Batch 2 material. SDS-PAGE examination of the PMT-active fractions showed enhancement of proteins in the 50-60 kD region. The molecular weight of PMT has been determined by gel permeation studies to be approximately 60 kD. Other minor bands were observed in the 40 and 33 kD region. Portions of the PMT-active fractions were pooled, concentrated, washed and subjected to agarose IEF. Coomassie Blue stained bands in the pH region associated with PMT-activity were further subjected to SDS-PAGE. Silver-staining of the SDS-PAGE gel showed an apparent band in the 60 kD region in this examination (5). Batch 2 material was also fractionated by conventional flat plate "native" polyacrylamide gel electrophoresis (2,5). Gel

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slices were assayed for PMT-activity and SDS-PAGE examination. Protein bands were observed in the 60 kD, and 30-40 kD regions for the PMT-active gel slices. (2)..

SS-DNA was released from a number of phagemids (7). The DNA sequences have been obtained for approximately 24 clones (100-150 base pairs) (6). Restriction mapping experiments have begun on several overly expressed tobacco root clones (6).

C. Plans: Continue to prepare partially purified PMT for further research. Elute PMT active IEF bands and resolve PMT by second dimensional gel electrophoresis (polyacryamide gels). Subject batch material to other chromatographic methods for the further purification of PMT. Determine the best method to obtain sample for protein sequencing. Continue comparative studies of protein assays using the KL and PM methods. Continue release of ssDNA for sequencing and attempt to sequence ssDNA. Continue restriction mapping. Prepare total RNA from tobacco plants for differential hybridization studies comparing young versus mature roots.

D. References:

1. Lyle, J. Notebook No. 8856, p. 118.
2. Turner, D. Notebook No. 8973, p.14.
3. Davies, S. Notebook No 8907, p. 197.
4. Yu, T. Notebook No. 8848, p. 176.
5. Nakatani, H. Notebook No. 8384, p. 143.
6. Wahab, S. Notebook No. 8855, pp. 185-186.
7. Malik, V. Notebook No. 8899, pp. 99-100.

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PROJECT NUMBER: 2106
PROJECT TITLE: Cigarette Performance and Design
PROJECT LEADER: R. W. Dwyer
PERIOD COVERED: May, 1990

I. PUFF COUNT CONTROL (D. Newman & B. Dwyer)

A. **Objective:** Determine the effects of wood-pulp cigarette wrappers on puff count.

B. **Results:** Six cigarette designs were fabricated using wood-pulp wrappers at various permeabilities and citrate levels; a companion set was fabricated with similar permeabilities and citrate levels using conventional flax papers.

C. **Conclusions:** Determinations of puff count, static burn times, and mass burn rates indicate that there are no significant differences in these properties between wood-pulp and flax papers.

II. FILTER PAPER ACETYLATION (D. Simpson)

A. **Objective:** Acetylate the surface of paper filters in order to provide the filtration efficiencies of paper and the subjective character of cellulose-acetate tow.

B. **Results:** In the laboratory, we have demonstrated that the paper used to make paper filters can be acetylated by either esterification or trans-esterification. Direct esterification with acetic anhydride and an acid catalyst appears to provide the best product in the laboratory. It was also found that rinsing the resultant product with ethanol rather than water provides a material with greater mechanical strength and a high degree of substitution. Subjective evaluations of cigarettes made with paper filters and acetylated-paper filters showed a significant advantage for the acetylated samples.

C. **Conclusions:** The major problem in implementing this program has been finding a source of acetylated paper. Paper maunfacturers are reluctant to work with the required chemicals, and chemical companies are not set up to treat bobbins of paper.

D. **Plans:** We are in the process of arranging a secrecy agreement with James River Co. in order to pursue this idea with them. We are also looking into the feasibility of setting up a pilot-scale acetylating facility inhouse.

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PROJECT NUMBER: 2500
PROJECT TITLE: Fundamental Chemistry
PROJECT LEADER: J. I. Seeman
PERIOD COVERED: May, 1990

I. INORGANICS AS NOVEL TOBACCO MATERIALS ADDITIVES (Fournier, Kallianos, Paine, Podraza, Secor, Seeman)

A. Objective: To develop inorganic materials for novel applications for reduced sidestream, for burn-rate modification, enhanced subjectives in cigarettes and for required properties in novel smoking materials.

B. Results and Plans: To develop a commercially viable sol-gel route to mag carbonates, we are examining the reaction of hydromagnesite [$Mg_5 (CO_3)_4 (OH)_2 \cdot 4\text{-}5H_2O$] with CO_2 in water. We have been successful in developing a small-scale procedure that goes through a gel state and provides particles of very small size. Smoking studies have been completed on a number of mag carbonates, and the results are extremely encouraging with many giving excellent SS reduction and a few with extremely high subjective ratings. This includes samples obtained in our contract work with Prof. D. Schleich based on carbonation of $Mg(OH)_2$. Efforts are underway to prepare materials having smaller particle sizes using technology which is commercializable.

Inorganic filler prepared by mild hydrothermal treatment of commercial hydromagnesite and $Mg(OH)_2$ gave paper which resulted in excellent SS reduction; resmoking of these cigarettes is in progress. Papers made from 10% of a "mag carbonate" prepared by the slow addition of a potassium hydroxide/carbonate solution to a sonicated solution of magnesium acetate were sized with K_2 Succ and resmoked.

A magnesite sample doped with five molar percent calcium has been prepared hydrothermally and a "mag carbonate"/$CaCO_3$ sample has been prepared by adding a potassium hydroxide solution to an excess of a magnesium/calcium acetate solution under sonication followed by the addition of a potassium carbonate solution. The calcium level of the second sample was adjusted to give 20 weight percent of $CaCO_3$ in the final product with the goal of improving ash integrity. Both samples will be submitted for papermaking.

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The synthesis of magnesite in the 2-liter stainless steel hydrothermal reactor has been started to determine impurity levels of heavy metals for production applications. This work should be completed in the next couple of weeks.

Analytical data for the matrix designed experiment using magnesium methyl carbonate is being evaluated. To date, the analytical data collected on all samples includes thermal analysis, FT-IR, particle size measurements, and SEM data. A sample from each of the experiments has been submitted for hand-sheet preparation. Several samples are being sent to VPI for Dr. Zallen to evaluate by Ramam spectroscopy. Vicki Baliga has examined several hand-sheets prepared using fillers from the matrix study. Nesquehonite is not present in the paper. Additional papers are being examined. The most promising filler, based on SS reduction properties, was prepared in a replicate run by an independent member of the group and excellent replication was observed.

Significant efforts are underway evaluating possible equipment purchases, including an FT-IR, a Zeta Potential Monitor, quadrapole MS, and various particle sizers. Individuals within the group and others are participating in these considerations.

An attempt has been made to prepare a solid solution of K-NH_4 magnesium carbonate from the reaction of hydromagnesite, and NH_4HCO_3. A single phase appears to have been formed, and analytical data is being obtained. It is hoped that a high-surface area amorphous K-containing mag carbonate will be achieved. We have also attempted to coat magnesite [$MgCO_3$] with calcite by reacting $CaCl_2$ with $KHCO_3$; calcite is generated slowly in a highly crystalline form; analyses are in progress.

II. REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS (Howe, Paine)

A. **Objective:** To develop techniques to remove selectively nicotine and other alkaloids from aqueous tobacco processing fluids.

B. **Results and Plans:** A special report entitled "The Removal of Nicotine from ART Monopotassium Citrate Solutions by Sulfonated Polystyrene-Divinylbenzene Ion Exchange Resins" has been issued. This report details the successful bench scale selective separation. An interim status report on the screening studies is being written.

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In collaboration with R. Southwick, C-18 Silica gel (C18), styrene-divinylbenzene copolymers (S-DVB) and polystrene (XAD) absorbents all function similarly to absorb the nonionic neutrals from the acidic BHPP water column solution. The S-DVB and XAD are preferred for pH stability reasons. Several large batches (>20 L) of solutions have been processed to provide 35 g of neutrals for isolation/identification/odor profiling and for spraying onto post-ART filler.

When the nicotine-laden aqueous filtrate obtained after removal of the neutrals (*cf.* above) is made basic and treated with fresh XAD or S-DVB absorbent, most of the alkaloids are removed. The nicotine can be obtained in a concentrated form (suitable for disposal) via an alcoholic wash.

III. MISCELLANEOUS (Howe, Paine, Secor)

A. **Results:** Microanalysis of the vanillin- and ethylvanillin-incorporated hydrotalcite suggest that the material is hexahydrated. A series of 34 BHPP-ART samples were processed for gc analysis by ARD. A number of substrates were prepared and delivered to E. R. Bernstein for cluster-jet spectroscopic analyses.

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PROJECT NUMBER: 2501
PROJECT TITLE: Smoke Chemistry
PROJECT LEADER: R. A. Comes
PERIOD COVERED: May, 1990

I. SIDESTREAM SMOKE CHAMBER

A. Objective: Operate an environmentally controlled chamber to measure selected components of sidestream smoke.

B. Results: The majority of the time during this reporting period has been spent on multiple chamber runs to assist personnel of the Biochemical Research Division. Experiments are in progress to address questions raised by some of the analytical data generated under certain conditions and with certain trapping procedures.

An instrument has been received to monitor CO and CO_2 from mainstream smoke in conjunction with the normal chamber sidestream operation. It was designed to our specifications to measure CO_2 and CO in the 0-10% range. Instrument set-up and calibration is in progress.

The multigas calibration/zero-air system has been installed. The system design provides required levels of ozone, carbon monoxide, non-methane hydrocarbons, sulfur dioxide, nitric oxide and nitrogen dioxide to perform zero, span checks and multiport calibration of our current Thermo Environmental instruments. A memo has issued describing the system set-up and operation.

A substantial effort has been carried out in conjunction with personnel of Hewlett-Packard to attempt to resolve the discrepancy between chamber nicotine values from the 5890 gc in the chamber lab and the 5880 gc on the fifth floor. The problems seem to be due to differences in the detection of the baseline in the two integrators which are made more noticeable by differences in the matrices of the standards and the samples. These differences are apparently detected with the different/more sensitive integration algorithm for the 3396 integrator on the 5890 gc. This anomaly will be further characterized and steps taken to make allowances for it in the analytical method.

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C. **Plans:** Chamber runs will continue to assist members of the BCR Division in their chamber related studies. The mainstream CO_2 /CO monitor will be operated in conjunction with normal sidestream chamber runs to investigate its utility in this regard. Other chamber runs will be carried out as required. Documentation of sample operating procedures continues.

II. SIDESTREAM SMOKE

A. **Objective:** Conduct studies on sidestream smoke including: development of methods for collection and analysis of sidestream semivolatiles and gas phase; visibility determinations; analysis of selected materials relating to sidestream odor and irritation; development of proprietary products.

B. **Results:** A system has been installed to investigate the visible sidestream smoke plume. A video camera photographs the sidestream plume throughout the static smoking process. The tape is then reviewed and the location of the visible sidestream smoke is noted in regard to the char line. Interesting observations have been made on selected cigarette models relative to the total smoking process. Current models under investigation are the control, $Mg(OH)_2$ wrapper and dual wrap $CaCO_3$ cigarettes used for the Analytical - Subjective studies.

Several smokings were carried out on the prototype CORESTA smoking machine to collect samples of mainstream and sidestream TPM in support of the sidestream odor modification program. Samples were collected under normal smoking conditions, the pads weighed and then turned over to the Analytical Research Division for determination of parent, decomposition product and/or release compound transfer to mainstream and sidestream smoke.

The new laser for the single port monitor was received and installed. Shortly after installation, the monitor began to behave erratically. Multiple attempts to determine the cause of the problem were unsuccessful. A new cell was made in a configuration to attempt to lessen the condensation of smoke droplets on the cell windows. This has, at least temporarily, solved the stability problem.

The Texmar concentrators used for some time to adsorb and desorb gas phase from cigarette smoke have proved to be inherently unsuitable for this analysis. They have been scrapped and parts from them have been used to construct a homemade apparatus for this work. Construction and testing are in progress.

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C. **Plans:** Continue investigation of the sulfur detector and attempt to determine differences in the sulfur containing compounds present in sidestream and mainstream gas phases from selected cigarette models. Assist in maintenance and construction of instruments to measure sidestream visibility.

III. MISCELLANEOUS

1. A series of samples was subjected to pyrolysis gc/ms at 200°C, 300°C and 600°C in support of the sidestream odor modification program. They included neat vanillin release compounds, cigarette papers onto which vanillin release materials had been coated vs untreated controls and samples of cigarette papers to which vanillin release materials had been applied in the sideseam adhesive. Gc/ms analyses were conducted in support of the Fundamental Chemistry Project. Modifications have been made to the system pyrolysis unit to facilitate sample pyrolysis in air. Memos will be written in support of all of these analyses.

2. In cooperation with the Computer Applications Division, a pc-based Perkin-Elmer Nelson Turbochrom II chromatography data system is being prepared and will be installed in the fifth floor lab for evaluation as a replacement for the PE LIMS system.

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PROJECT NUMBER: 2520
PROJECT TITLE: Flavor Research
PROJECT LEADER: Y. Houminer
PERIOD COVERED: May, 1990

I. COMMERCIALIZATION OF GMC AND POLYMIC

A. **Objective:** To find chemical manufacturers who are capable of producing large quantities of GMC and polyMIC.

B. **Results:** The production of batch 004 of GMC was carried out at Lee Labs. Approximately 215 pounds of product was produced (expected yield - 290 pounds). The lower yield was most probably due to losses associated with carbon treatment. In an attempt to obtain the same low color as in batch 003, the product was treated twice with carbon. The total amount of carbon used for this batch (two treatments) was of the order of 40 kiligrams. With batch 003, a different filter was used and the amount of carbon was closer to 10 kilograms. In the future this excessive carbon treatment will be eliminated as it has been found that the yellow color obtained with GMC is not readily removed with carbon treatment. Analytical data is currently being determined on this material.

After the production of batch 004 we used the reactor to dilute approximately 30 gallons of 77% GMC (batch 003) to 61.5% GMC with absolute ethanol. Dilution went well with only minor heating needed to dissolve the GMC. It was noted that on storage for about 6 months, the color of the solution had an increased yellow cast.

An outline of the current status of the PolyMIC project was received from Callery Chemical. Work with potassium enolates is showing some success. Work with quaternary ammonium enolates has not been successful.

II. FLAVOR RELEASE TECHNOLOGY

A. **Objective:** To investigate the synthesis and pyrolysis of various flavor release systems for use in new or improved products.

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B. Results: The large scale production of CR-2910 at Aldrich has been completed. A total of 190 lbs have been produced. Forty pounds were delivered to PM. The purity of the material is 99% by HPLC.

A research contract has been given to R.S.A. to develop a method for the synthesis of Vanillin Chloroformate. Method is to be suitable for large scale production of this material.

Methyl 4,6-O-(4'-t-butoxycarbonyloxy-3'-methoxybenzylidene)-α–D-glucopyranoside (t-BOC-Aromatek-215) was prepared from methyl 4,6-O-(4'-hydroxy-3'-methoxybenzylidene)-α-D-glucopyranoside (A-215) and di-t-butyl dicarbonate in high yield. We are in the process of attempting to crystallize this material. A sample of the crude material was delivered to Dr. Ted Sanders for stability evaluation.

Also prepared was divanillyl carbonate from vanillin and triphosgene. The material is only slightly soluble in ethanol at room temperature, but unlike the divanillyl oxalate, this material can be recrystallized from a variety of solvents. Preliminary pyrolysis GC/MS showed that at 300°C, the material pyrolyzed to a large extent to give carbon dioxide and vanillin.

III. FLAVOR CHEMISTRY

A. Objective: To obtain flavors for subjective evaluation and odor profiling. To isolate and identify tobacco components which are sensorially significant.

B. Results: We continue our work on the recovery of organic components from the ART process water column effluent. Different material, such as revered-phase silica gel (C-18), are being tested.

About 60 liters of effluent were used to isolate organics for subsequent fractionation and identification. A sample of 35g material has been isolated and was fractionated on silica-gel to give eight fractions. Odor profile and subjective smoking will be used to identify the best fraction.

Work continues on the removal and recovery of nicotine from XAD-4 resin (polystyrene).

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PROJECT NUMBER: 2525
PROJECT TITLE: Paper and Tobacco Research
PROJECT LEADER: G. H. Bokelman
PERIOD COVERED: May, 1990

I. SIDESTREAM REDUCTION (S. Baldwin, S. Tafur, G. Bokelman, J. Kang, G. Newell, P. Suiter and B. Rogers)

A. Objectives: (1) Prepare and evaluate, for reduction in sidestream smoke, paper handsheets that contain additives and/or inorganic fillers with different physical and chemical properties and (2) evaluate machine-made cigarettes containing cigarette papers produced at the University of Maine or by our vendors.

B. Results: A new laser was installed by Doug McRae on the single port SS (sidestream) visibility instrument. To achieve satisfactory operating conditions, a beam expander was also installed on the instrument. The cell was re-designed to give improved smoke flow characteristics and flow rate was optimized for the new cell. Following completion of these instrument modifications, routine analyses of handmade cigarette samples were resumed.

Additional data continue to verify the fact that sidestream reduction is enhanced when certain organic acids are combined with an alkali metal salt and used as a fluxing agent on paper. The sidestream reduction obtained exceeds that obtained from the acidity alone (as observed previously with H_3PO_4) or with the alkali metal salt at equivalent or higher levels. All acids are not equally effective, even after compensating for molecular weight. The reasons for the differences are not fully understood at this point. Both mono and dicarboxylic acids are effective. Citric acid is much less active than would have been predicted, based on equivalents of acid added. This finding may be due to the chelating ability of the citric acid which could weaken the ash. These results represent indirect evidence which support the hypothesis that melting of the acids, in addition to their acidity, contributes to the overall effectiveness. The melting is presumed to strengthen the ash.

Examination of magnesium-containing papers prepared with fillers synthesized by members of Project 2500 or obtained from N.Y. Poly. was continued. Comparisons were made of mixed composition materials prepared from various forms of $Mg(OH)_2$ (paste, freshly precipitated, and commercial powder) under different reaction conditions. Virtually all the materials gave good SS reductions with no obvious preference for the starting form of $Mg(OH)_2$. Differences in SBT

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(static burn time) were noted. Many of the materials at a 30% level in the paper give >11 min SBT, which is longer than desired. Incorporation of lower levels of these new fillers along with the addition of some $CaCO_3$ in the paper should help ash appearance and SBT. Preliminary subjective screening of handmade cigarettes made with a number of these papers is in progress.

Other papers prepared with synthetic fillers included a magnesite product made from $Mg(OH)_2$ paste which performed well (62% SS reduction) and gave encouraging subjective results. A "$MgCO_3$ " sol-gel material made from hydromagnesite also looks promising. Several mixed materials prepared from $Mg(OAc)_2$ also were examined.

A series of handsheets made by Ecusta that incorporated mined magnesite samples from China and Greece were used to prepare handmade cigarettes. SS analyses indicated good SS reduction but the impurities in these particular mined materials (manganese, silicon) probably make them unacceptable candidates for cigarette papers.

An attempt was made to improve the ash flakiness seen with Canadian Baymag A magnesite papers by inclusion of various levels of $CaCO_3$. As $CaCO_3$ level increased, ash firmness improved but at the expense of SS reduction performance. In another experiment, the addition of a small amount (5%) of magnesite to a high basis weight $CaCO_3$ paper was shown to give the desired "slightly less firm" ash.

Preliminary examination of a succinic acid sizing on magnesite and hydromagnesite papers suggested a slight improvement in SS reduction, but with a longer SBT. Other acids will be examined.

Combinations of magnesite and a precipitated derivative of dolomite (prepared by Pfizer) were tried as paper fillers. It was found that the use of such mixed fillers does not appear to offer any advantage.

A combined sizing of galacturonic acid with KH_2PO_4 applied to medium and high basis weight $CaCO_3$ papers was shown to give good SS reduction, even with lower KH_2PO_4 levels.

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Medium basis weight papers sized by Kimberly-Clark with sodium fumarate were analyzed. This additive does not appear to offer any particular advantage over KH_2PO_4, except that it may be somewhat more effective in SS reduction at relatively low levels of addition.

Work continues on the preparation of a $CaCO_3$ -encapsulated $Mg(OH)_2$ filler. Ten additional samples have been prepared. Calcium and magnesium levels indicate some problem with three batches in which the magnesium content seems too low. Replicate analyses are being performed to determine whether the problem is sample uniformity or incorrect addition of starting material. TGA analyses are pending. Upon receipt of these data, additional samples will be submitted for papermaking.

Ecusta has completed analysis of a series of handsheets designed to optimize the magnesite paper with respect to basis weight, porosity, and magnesite content. Upon receipt of the data, however, it became apparent that the porosity specifications had not been met. This led to a separate study to correlate porosity measurements made at PM and Ecusta. Following completion of that study, five additional handsheets were requested for Ecusta to prepare and analyze. Upon receipt of these additional data for SSTPM, puff count, and MSTPM (and ash), multiple regression analyses will be performed to optimize the three independent variables in preparation for a production run on Ecusta's paper machine in July.

Work on the effect of acids and bases on cellulose pyrolysis with high basis weight paper has begun. This research is being conducted under contract with Dr. Helena Chum at the Solar Energy Research Institute. Eventually this work will include the use of isotope-enriched cellulose prepared at PM.

The fabrication of handmade cigarettes using a roller maker (Rizza) obtained from Ecusta was examined. The tobacco for making cigarettes with the roller is obtained by carefully removing the paper from machine-made cigarette rods and maintaining the shred density and orientation. Preliminary work has shown that well-packed handmade cigarettes can be prepared by this method and SS visibility results are similar to those obtained on handmade cigarettes made by adding tobacco with a Supermatic maker to preformed paper tubes. Additional comparisons will be needed to determine if we can switch to the roller maker. The method potentially can save time in handmade cigarette fabrication and may improve the cigarette-to-cigarette variation inherent in handmade cigarettes.

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BET surface area analyses were continued this month by John Kang with the examination of the effect of heat on a hydromagnesite/K, citrate (3:1) mixture. The mixture became somewhat granular upon heating, which suggested that fluxing might be occurring. The resultant surface area, however, increased compared to that typically found with hydromagnesite alone. Examinations of other filler/additive and paper/additive combinations are in progress. A programmable furnace was acquired and installed to facilitate this work. Initial comparisons have shown similar results are obtained with the new furnace vs. the tube furnace previously utilized.

A course on simplex optimization offered by *Statistical Designs* of Houston, Texas, was attended by Sherry Baldwin on May 14 and 15. The techniques described were quite useful. One reason for taking this course was to determine whether or not better ways might be found to optimize reduced sidestream papers. It turns out that the statistical regression techniques we are currently using are the most appropriate for our purposes. However, the simplex techniques may be ideal for studying other relevant problems, such as optimization of sol-gel yields. An additional point which was discussed was the use of *desirability functions*. These functions can be used to incorporate subjective attributes into a regression and simultaneously optimize several desirable parameters. The use of one of these functions will be attempted in the magnesite regression as a means of simultaneously optimizing several factors, including subjective ones such as ash quality.

For the month 33 handmade cigarette models were prepared for the reduced SS program. Two sets of handmade cigarettes (20 cigarettes each) made from cigarette paper handsheets incorporating vanillin-release compounds (CR 2905, CR 2900) were prepared for analytical studies. Four handmade models were prepared from single component Japanese and U.S. bright fillers. In addition to the tests of selected cigarette models for establishing optimal operating conditions of the laser single port instrument, routine analyses of 37 models were conducted.

C. Plans: Sixteen more models will be prepared to study the interaction of organic acids with alkali metal salts. Additional samples of magnesium hydroxide particles encapsulated with calcium carbonate will be generated in-house and analyzed. Paper handsheets containing various samples of synthetic "$MgCO_3$" will continue to be evaluated. Cooperative efforts with Ecusta to develop the commercial potential of magnesite papers will be continued. Finally, the paper database will be updated.

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II. ESTIMATION OF CIGARETTE BLEND COMPOSITION (G. Bokelman, J. Stimler and General Analytical)

A. **Objective:** Examine the blend compositions of Keith (a new product entry by JTI) and Caster.

B. **Results:** It appears that both the Keith Mild KS and the Keith Slim LS may be produced using the same blend (1). However, this common blend is very different from a "typical" blend used by JTI. Specifically, the Keith brand products are both much higher in burley lamina and Oriental leaf and much, much lower in stems than the "typical" blend used by JTI in the production of Mild Seven. The data indicate that Keith may be designed to use an all lamina blend.

It appears likely that Caster and Caster Milds are designed to have the same blend. The blend composition data (2) show that, compared to a typical U. S. commercial cigarette, Caster has a very high bright lamina content, a low burley lamina content, an extremely low Oriental leaf content and a high stem content. The blend compositions of Mild Seven and Caster are rather similar.

It was suggested that the PM Super Lights blend might be a good starting point for developing a "Caster-like" blend. The main difference is that the PM Super Lights blend has much more Oriental leaf and a little less of all the other tobacco components compared to Caster.

C. **Plans:** We will be examining a number of cigarette brands produced in the U. S. for sale in Japan, as requested by Judy Smith.

D. **References:**

1. Bokelman, G. H. and J. O. Stimler, memo to C. R. Lambert, Jr., "Blend Composition Analyses of Keith Mild KS and Keith Slim LS," May 18, 1990.

2. Bokelman, Gordon and Jack Stimler, memo to Judy Smith, "Blend Composition Analyses of Several Cigarette Samples," April 27, 1990.

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III. GREENHOUSE STUDIES & SUPPORT ACTIVITIES (R. Bass, G. West and G. Newell)

A. **Objectives:** Maintain the R&D greenhouses, conduct plant research studies, provide greenhouse-grown tobacco materials for support of other R&D programs and provide requested assistance for special projects.

B. **Results:** The hydroponically grown Burley 21 plants of Group 23 were harvested. These 52 plants yielded roots with a total weight of 19.4 kg. The hydroponic system was set up for Group 24 and transplanting of the seedlings was completed.

A special request was received from Project 1904 for the hydroponic production of tobacco and tomato plants in the Greenhouse. The appropriate materials have been seeded and will be grown as requested.

Greenhouse tours and tobacco displays were provided for visitors from PM Corporate Affairs, Kraft General Foods and PM Latin America.

As requested, tobacco plants were provided for display purposes at PMIS, York Engineering, the Annual PM Stockholders' Meeting and the Board of Directors Meeting.

^{14}C-radiolabelled materials were supplied for the fabrication of 21 cigarettes as requested by Andy Frisch.

The regular greenhouse plant production activities were completed including the formulation of 660 gallons of nutrient solution, several insecticide applications, watering, transplanting, topping, suckering, trimming, and seeding of Burley 21, Speight G-28, Kentucky-326, N. rustica, N. glauca, Oriental tobacco and *Better Girl* tomatoes.

C. **Plans:** Maintain plant production schedules. Inspect and monitor plant and environmental conditions on a daily basis.

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PROJECT NUMBER: 6502
PROJECT TITLE: Environmental Tobacco Smoke
PROJECT LEADER: C. E. Thomas
PERIOD COVERED: May, 1990

MAINSTREAM AND SIDESTREAM SMOKE STUDIES

A. **Objective:** Develop a method, using the tunable diode laser system (TDL), for the determination of carbonyl sulfide (COS) in MS and SS tobacco smoke.

B. **Results:** Three COS infrared absorption lines were found to be free of interferences from major gases in cigarette smoke. The wavenumbers of the lines selected ranged from 2050 to 2090 cm^{-1}. Ten IM13 cigarettes were smoked on each line. An MG Industries standard of 271 ppm COS diluted with nitrogen to 36.7 ppm was used for calibration purposes. The averaged total MS COS deliveries for IM13 cigarettes determined on each line were : 30.1 ± 1.6 µg/cigt, 31.1 ± 2.6 µg/cigt, and 30.3 ± 1.1 µg/cigt.

C. **Conclusions:** The accuracy of the COS measurements using TDL spectroscopy has been evaluated using three different infrared absorption lines. The MS COS delivery for IM13 cigarettes averaged 30.5 ± 1.9 µg/cigt (RSD @ 2σ = 12%).

D. **Plans:** MS COS deliveries will be determined for Kentucky and FTR reference cigarettes. Additional studies will examine the effects of cigarette blend components on MS COS deliveries.

E. **References:**

Lipscomb, J., PM Notebook #8914, pp.73-78.

A. **Objective:** Develop a method for primary amines in MS and SS tobacco smoke.

B. **Results:** An HPLC procedure for primary amines in tobacco smoke has been developed. The method collects the amines from gas phase MS and SS smoke on acid-treated silica gel collection tubes. The amines are trapped as their hydrochloride and solvated with 2 ml of 0.1N HCl. Methyl and ethyl amine are derivatized by o-phthalaldehyde and mercaptoethanol, separated on a ODS-2, 25-cm, C-18 column and quantitated by fluorescence detection of the derivatized species. The detection limit of the method is 0.1 µg/cigt. Methyl amine was quantitated in the SS of 100% Bright, Oriental, and Burley cigarettes. The concentrations were 80 ± 8, 52 ± 5, and 125 ± 10 µg/cigt

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respectively. MS and SS methyl and ethyl amine were also determined for IM13. For methyl amine the MS was 7.0 µg/cigt and the SS was 77. µg/cigt. Ethyl amine was detected (less than 1.0 µg/cigt) in the MS and 18 µg/cigt. was found in the SS gas phase.

C. **Conclusions:** A method has been developed for MS and SS primary amines. The method will be used in support of the low sidestream project and studies on analytical /sensory correlations of sidestream smoke.

D. **Plans:** Work will continue on determining the accuracy and precision of the method. 100% blend component cigarettes will be studied to see how the blend of a cigarette affects the delivery of the low molecular weight primary amines.

E. **References:**

Randolph, H., PM Notebook #8799, p.86.

A. **Objective:** Determine the effect of KH_2PO_4 and KSuccinate treated cigarette papers on MS gas phase smoke.

B. **Results:** The MS gas phase smoke from cigarettes made with KH_2PO_4 or KSuccinate treated paper was analyzed by FT-IR spectroscopy using a Digilab FTS-60 spectrometer. Cigarettes made with KC paper P9XE treated with 13.3% KSuccinate (X8D0DP), 4.0% KH_2PO_4 (X8D0DX), and 18.7% KH_2PO_4 (X8D0EB, X8D9ECW) were analyzed. The tobacco was Marlboro filler J9CBS, without aftercut, for samples DO, DP, DX, EB, and Marlboro filler J9BUS, without aftercut, for sample ECW. Only comparative, qualitative results were obtained. The gas phase absorption line peak areas for twelve gases from the KH_2PO_4 treated cigarettes were compared to the values from the 0% base sheet paper cigarettes (X8D0DO). The P9XE paper reference cigarette was compared to a P8LX paper reference cigarette made with Marlboro filler with aftercut (X8D9CRI).

C. **Conclusions:** The concentrations of all 12 of the MS gas-phase components were lower in the P9XE paper cigarettes. This difference could be due to either the aftercut, the paper, or both. No clear trends were evident in the data from the P9XE paper cigarettes.

D. **Plans:** A procedure for determining the concentrations of gas phase components of SS smoke, using FT-IR spectroscopy will be developed and used to analyze the KH_2PO_4 modified cigarettes.

E. **References:**

Koller, K., PM Notebook #8870, pp. 91-93.

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PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations/Methods Development
PROJECT LEADER: D. F. Ingraham
PERIOD COVERED: May, 1990

I. AMBROSIA

A. Objective: Provide analytical support for the development of compounds targeted as potential sidestream odorants

B. Results:

Methods were developed to determine trichloroacetic acid, dimethylformamide, vanillin, and di-*tert*-butyl dicarbonate in VANIL-3 for specification purposes. These methods were used to determine the levels of these compounds in samples produced by Aldrich. The purity of the VANIL-3 and the amount of vanillin present were also determined.

Analysis of VANIL-3 on cigarette paper showed about a 90% loss of the compound after storage of cigarettes containing VANIL-3 (on the paper) in the desert room for one week. Only a small amount of this loss could be attributed to its transfer to filler.

Sidestream and mainstream smoke samples obtained from cigarettes containing VANIL-3 on the paper were analyzed for vanillin and VANIL-3. Smoke samples from cigarettes containing Aromatek-245 were also analyzed to determine the amounts of Aromatek-245 and α-hexylcinnamic aldehyde. The results were reported to R. Comes.

C. Plans: Continue to support this program as needed.

D. References:

1. Yang, S. S., "Determination of the Cigarette Paper and the Smoke Products of CR-2910," memo to R. Comes, May 7, 1990.

2. Yang, S. S., "Determination of the Smoke Products of Aromatek-245," memo to R. Comes, May 17, 1990.

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II. RESPONSE TO ANALYTICAL REQUESTS

A. Objective:. To provide analytical support to R&D and Operations personnel and projects.

B. Results:

Analyses and investigations by project personnel during the month of May included:

Several customer complaint samples were analyzed.

Quantitative and qualitative analyses of various packaging materials were performed for Rick Dunaway and Packaging Technical Services.

Several dirt samples from 20th Street were analyzed for volatile organic compounds. Two of the samples contained propionic acid, propylene glycol, acetic acid, and propionic acid esters of PG. In a third sample, only PG was observed.

The amounts of ethanol present in production samples of MGC (Lee Labs) were determined, in addition to the concentration of MGC in the solution.

The amount of ethylvanillin glucoside on the paper of Horizon (RJR) cigarettes was determined.

The amounts of various phenolic compounds were determined in smoke samples from Caster cigarettes.

The amount of propylene carbonate (PC) in cigarette rods containing target levels of 2% and 4% was determined. Results showed the amounts of PC to be significantly lower than the targets. The individual cigarette results were forwarded to M. Parrish to determine the uniformity of application of the PC.

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PROJECT NUMBER: 6902
PROJECT TITLE: Biochemical Special Investigations
PROJECT LEADER: B. D. Davies
WRITTEN BY: E. A. Crockett
PERIOD COVERED: May, 1990

I. NICOTINE SPECIFIC MONOCLONAL ANTIBODY

A. **Objective:** Develop a nicotine monoclonal antibody based enzyme-linked immunosorbent assay (ELISA) for the determination of nicotine concentrations in samples of interest to PM.

B. **Results:** Several experiments were conducted to determine the causes of ELISA variation and, if possible, reduce it to acceptable levels. One such experiment was conducted to examine the variation contributed to the overall ELISA assay by the optics of the plate and the plate reader. A solution of nitrophenol was prepared so that a 100 µl aliquot had an absorbance of ~1.0 OD as measured by the plate reader. A 100 µl aliquot of this solution was individually pipetted, using a Rainin pipet gun, to all 96 wells of one of the standard microtiter plates. This plate was then placed in the microtiter plate reader for quantitation of the absorbance of all wells. The percent relative standard deviation (%RSD) for these wells was 1.28%. This indicates that when the solutions are pipetted by hand, the plate and plate reader are not the major contributor to variation in the assay. Another experiment was performed to investigate the variation contributed by different types of pipettes. An Eppendorf repipettor, Rainin pipet, and a Hamilton syringe were used at various stages in the ELISA assay. A difference was observed between pipet types. The repipettor gave a %RSD of 14.4 which is unacceptable. The %RSD for the Rainin and Hamilton were found to be 11 and 12.5, respectively. Since the Hamilton syringe increases the amount of time that it takes to apply the samples, the Rainin pipet will be used in the ELISA assay. A similar experiment was run using only the Eppendorf repipettor and the Rainin pipet. However, the microtiter plates were shaken during incubation in order to facilitate diffusion of the antibody in the microtiter well. The %RSD increased for both types of pipets. The %RSD for the Eppendorf repipettor was 14.7 and the Rainin pipet 14.1. In an attempt to look at the variation between tobacco extracts the BD1 - BD6 samples were run in an ELISA in triplicate. Within triplicates of the same sample the %RSDs were as follows: 4.8 - 10.1 (BD1); 4.2 - 11.5 (BD2); 5.0 - 15.6 (BD3); 5.2 - 12.0 (BD4); 3.8 - 8.5 (BD5); and 7.6 - 11.3 (BD6). When the data from the triplicates were combined the %RSDs were as

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follows: 9.1 (BD1); 9.4 (BD2); 9.8 (BD3); 14.8 (BD4), 8.0 (BD5); and 15.4 (BD6). And, when all the data were combined as one set the %RSD was 19.2. However, these data were obtained using the Eppendorf repipettor which has been shown to contribute to the variation found in the ELISA assay.

An experiment was run in which the liners to the extraction vial caps (in which the nicotine extraction is done) were placed in their normal vs an inverted position. A known amount of water was added to each vial and heated at 90° C for 24 hours. Afterwards each vial was weighed. The vials which had the liners placed inverted with the silicon side down toward the sample showed less water loss (0.02 grams) and less variation then the vials with the teflon side down (as much as 0.10 grams water loss).

C. **Plans:** Continue to assist with analysis of assay variance and possible assay modifications to reduce the variance. Plans include the investigation of other types of pipets (Eppendorf Multichannel electronic digital pipet and the single channel electronic pipet). Intra-well variation, plate to plate variation, and protein uptake will be examined by use of a variety of plates obtained from different manufacturers. The affect of shaking during incubation will be further investigated. The absorbance of the monoclonal antibody (BE2) will be increased from 1.0 to 2.0 OD and another monoclonal antibody (HA4) which has a 10-fold higher affinity than BE2 will be further characterized. D. Jones of the Product Evaluation Division will be performing further statistical analyses on the data.

D. **References:**

Davies, B. D. Notebook No. 8638, p. 180.
Crockett, E. A. Notebook No. 8933, pp. 112-152.

II. ADDITIONAL APPROACHES TOWARD PUTRESCINE METHYLTRANSFERASE (PMT) ISOLATION

A. **Objective:** Provide additional experimental approaches to assist in the effort to isolate PMT.

B. **Results:** A second batch of ^{3}H-Phenylethanolamine N-Methyltransferase (^{3}H-PET), photoaffinity labeled with and without S-Adenosylhomocysteine (SAH), was sent to Kendrick Labs for two dimensional sodium dodecyl sulfate-polyacrylamide gel electrophoresis (2D SDS-PAGE) analysis of labeled proteins. As in a previous

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experiment ~3 major proteins were labeled in the absence of SAH, but none in its presence.

A portion of this same ^{3}H-PET was serially diluted (1/2) and fractionated on one dimensional SDS-PAGE. Following an 18 hour exposure, the minimum number of counts which could be visually detected was ~500 dpm for this length of exposure.

The peak PMT-containing fraction from phenyl-Sepharose fractionation (PS-PMT) of root material performed by Project 1904 members, was concentrated and photoaffinity labeled (PAL). Three major protein bands (~30-39 kD, ~72kD and ~94kD) labeled in the absence but not the presence of SAH or putrescine.

A portion of affinity eluted, Batch 2, PMT-containing preparation was washed, concentrated, and subjected to PAL. Two major protein containing bands (~35-39kD and ~94kD) labeled in the absence but not in the presence of SAH or putrescine.

The results with the concentrated PS-PMT and Batch 2 PMT would seem to indicate that all of these bands contain proteins which are sensitive to the effects of putrescine. However, the concentration of putrescine used to block binding may be high enough to nonspecifically effect the labeling of proteins which are normally insensitive to the effects of putrescine.

A preliminary experiment was conducted to determine the feasibility of labeling PMT with ^{3}H-SAM in the absence of photolysis. The results of this experiment indicate that PMT can covalently bind ^{3}H-SAM under some incubation conditions.

C. **Plans:** An experiment will be conducted to examine the effects of varying the concentration of putrescine in the reaction mixture on the levels of ^{3}H-SAM incorporation into specific protein bands. Continue to label PMT containing fractions of increasing PMT specific activity. Continue to examine the feasibility of labeling PMT without photolysis.

D. **Reference:**

Dunn, R. L. Notebook No. 8721, pp. 156-176.
Davies, B. D. Notebook No. 8638, p. 180.

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PROJECT NUMBER: 6906
PROJECT TITLE: Biological Effects of Smoke
PROJECT LEADER: G. J. Patskan
WRITTEN BY: L. H. Thompson
PERIOD COVERED: May, 1990

I. GLUTATHIONE DEPLETION ASSAY (GDA)

A. **Objective:** To study the effect of cellular glutathione (GSH) levels on mutagenicity to establish the relevance of GSH depletion by CSC.

B. **Results:** A V79 mutation assay was conducted using MNNG under several modified treatment conditions: 1) a modified standard assay (treatment in Eagle's Minimum Essential Medium (EMEM) with fetal bovine serum (FBS) but with a cell density of $2X10^7$ cells/75cm² plate instead of $3X10^5$), 2) treatment in EMEM without FBS, 3) treatment in 150cm² flasks, and 4) trypsinizing and reseeding cells at lower densities ($2X10^5$ cells/plate) after treatment.

The results indicated that there was similar mutagenicity for treatments in EMEM with and without FBS (~ 45 and 48 mutants/plate, respectively). Also, trpsinization of cells after treatment and reseeding ($2X10^5$ cells/plate) resulted in ~ 54 mutants/plate. Cells treated in the 150cm² flasks produced ~ 115 mutants/plate. These colonies were larger and twice as many mutants/plate were produced compared to cells treated in 75cm² plates. This suggests that there is a direct relationship between cell density and the number of mutants. Cytotoxicity was only observed under the trpsinization treatment procedure (50% cell death). A second experiment is in progress to confirm all of the above observations. Any of the treatment methods applied here would be compatible with GDA protocol.

C. **Plans:** An experiment is in progress to test the effect of cellular GSH depletion on MNNG mutagenicity using the trypsinization method. Results will be reported next month. In addition, the modified standard procedure will be used if the mutagenicity assayed results are confirmed.

D. **References:** McCoy, W. R. Notebook No. 8925, pp.172-176.

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II. JB6 MOUSE EPIDERMAL CELL TRANSFORMATION ASSAY

A. Objective:. To establish the JB6 mouse epidermal cell transformation assay in our laboratory.

B. Results: The results from a plate-growth toxicity assay with RN CSC (bright baseweb) (0.1-15 ug/ml) were inconclusive because of unusually low cell counts for clone 41 (promotion-sensitive cells). A soft agar assay evaluating both frozen-processed and fresh (unprocessed) IT CSC at doses of 0.1-25 ug/ml showed no colonies on any test plates after a 3-week incubation period. A second experiment at higher concentrations (25-100 ug/ml) indicated toxicity on the RT101 (positive control) plates at the 50 ug/ml test dose. There were no colonies on the clone 41 test plates. An additional experiment is in progress evaluating 12-*O*-tetradecanoylphorbol-13-acetate (TPA), mezerein and frozen processed 2R1 CSC in both 10 and 20% serum. Although the results are incomplete, qualitatively, it appears that 20% serum may be necessary to support colony formation by 2R1 CSC. Another experiment with 2R1 CSC was begun involving a 4 day pretreatment of cells in flasks prior to the soft agar assay.

C. Plans: Quantitatively evaluate all the experiments in progress. Conduct a dose response experiment with 2R1 CSC in 20% serum agar medium.

D. References: Nixon, G. M. Notebook No. 8711, p. 155.

III. EPIDERMAL GROWTH FACTOR (EGF) ASSAY

A. Objective: To determine the effects of catechol-depleted CSC on binding in the EGF assay.

B. Results: Previous results using RTLC scanning indicated that the enzyme tyrosinase degraded catechol in 2R1 CSC. In a recent experiment, the effects of tyrosinase alone as well as catechol-depleted 2R1 CSC on 3T3 cells in the EGF assay were evaluated. The results showed that with tyrosinase treatment alone, EGF binding was increased. The results with catechol-depleted 2R1 CSC were inconclusive.

C. Plans: More experiments are planned in this area.

D. References: Vaughan, B. G. Notebook No. 8948, p.6

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PROJECT NUMBER: 6908
PROJECT TITLE: Smoke Condensate
PROJECT LEADER: A. H. Warfield
PERIOD COVERED: May, 1990

I. REMOVAL OF TSNA PRECURSORS

A. **Objective:** To develop methods for the removal of TSNA precursors from filler.

B. **Results:** It has been hypothesized that calcium incorporated in the tobacco matrix may be associated with precursors of MS TSNA, and that calcium removal may result in reductions of these precursors. To test this hypothesis, DBC Burley (Bu) has been washed with aqueous acetic acid (1*N*) and lanthanam chloride (0.1*N*) solutions in attempts to remove calcium. Analytical data from these experiments are incomplete.

C. **Plans:** Complete data analysis of the washed fillers and initiate mineral acid washes if the acetic acid and/or lanthanum washes prove to be ineffective at calcium removal.

D. **References:**

Haut, S. A. Notebook No. 8891, p. 82.

II. NITROSATING AGENT INHIBITORS

A. **Objective:** To reduce the levels of pyrosynthesized TSNA by interfering with the nitrosating agent(s).

B. **Results:** In an attempt to investigate the utility of a possible nitrosating agent scavenger, phytylmethyl oxalate (PMO) was added to a Bu filler which had been extracted with 5% ethanol in hexane to remove nicotine and preformed TSNA. PMO, obtained from Dr. Rett Southwick, is a known phytadiene release agent. MS TSNA delivered by the sample with added PMO showed no significant reduction in MS TSNA when compared with the control.

C. **Plans:** Obtain gas phase data from CTD to determine whether NO was reduced in the treated sample.

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D. References:

Haut, S. A. Notebook No. 8891, p. 82.

III. UNEXTRACTED NICOTINE STUDIES

A. Objective: To determine whether there is a correlation between unextracted nicotine (UN) in filler and the delivery of NNK in the corresponding MS smoke.

B. Results: Experiments were carried out in which various enzymes were utilized to convert Nic-Y in water-washed SCFE burley filler to water-soluble forms of UN. The water-soluble Nic-Y would then be analyzed as Nic-X. Therefore, the measure of a successful treatment would consist of a significant *increase* in Nic-X along with a corresponding *decrease* in Nic-Y relative to data from a control experiment in which the enzyme was left out.

An initial study was completed in which cellulase, β-glucosidase, and pronase E were used to digest the filler for 24 hr at 35°C, compared to water-treatment under the same conditions. Cellulase and pronase E gave higher Nic-X levels, but pronase E also gave higher Nic-Y levels. The Nic-Y level for the cellulase sample was not obtained due to experimental difficulties. Pronase E appeared to be the best choice. However, the increases in Nic-X were very small relative to the amount of Nic-Y found in the samples. An additional study was carried out in which proteolytic enzymes were compared to the same treatment minus the enzymes. The enzymes used were Nagarse Type VII, Protease Type XXI, Subtilisin Carlsberg, and Trypsin, as well as Pronase E. Nagarse and Subtilisin Carlsberg gave sigificant decreases in Nic-Y along with corresponding increases in Nic-X, with Nagarse providing the best results. Although these changes again were small relative to the levels of Nic-Y, the proteolytic enzymes appeared to be the most probable choices thus far for use in a future preparative attempt to isolate a water-soluble modification of Nic-Y.

C. Plans: Additional enzyme digestion experiments will be performed in attempts to optimize the liberation of Nic-Y for isolation.

D. Reference: Lambert, E. A. Notebook No. 8852, pp. 166-169.

E. Abbreviations/Definitions: Nic-X = hot water-soluble alkali-releasable nicotine. Nic-Y = hot water-insoluble alkali-releasable nicotine.

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IV. ANALYTICAL PROCEDURES

A. Objective:. To develop and maintain analytical methodology for TSNA or other compound classes relevant to the nitrosamine content of smoke or filler.

B. Results: Further work has been done on an analytical method for the determination of pseudo-oxynicotine (PsON). Thus far, attempts to derivatize the carbonyl group with 2,4-dinitrophenylhydrazone (DNPH) have been unsuccessful. Recently, this reaction was tried again using DNPH in acetonitrile, but this also failed to provide the derivative. When a solution of PsON hydrochloride was made basic and passed through a sulfonic acid solid phase extraction column, GC/NPD afforded four peaks, one of which corresponded to that observed when PsON was made basic, extracted and injected into the GC. This indicates that one approach may consist of direct analysis of PsON by GC without derivatization, although there appear to be many possibilities for further reactions if this approach is adopted.

C. Plans: Meet with selected individuals to formulate additional approaches to the analysis of PsON.

D. References:

Keene, C. K. Notebook No. 8946, pp. 51-52.

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PROJECT NUMBER: 6912
PROJECT TITLE: Tobacco/Smoke Relationships
PROJECT LEADER: R. R. Izac
WRITTEN BY: S. Drew
PERIOD COVERED: May 1990

I. CROSS SOLUBLES/BASE WEB (CHEMISTRY)

A. Objective: Modify tobacco solubles and evaluate the smoke chemistry of fillers from various treated solubles and the appropriate base web.

B. Results: The impaction trapped cigarette smoke condensate (IT CSC) from the silver nitrate model cigarettes has been prepared, stored and submitted for S/M assay testing. A hollow fiber membrane experiment designed to remove material on the basis of molecular weight was completed. Molecular weight cut-offs were 10kD, 50kD, and 500kD. An experiment using ultrafiltration in combination with ion exchange to modify tobacco solubles is being repeated. The effects of irradiation using a quartz mercury-vapor lamp on BuS1 are also being evaluated. An experiment using 30% hydrogen peroxide to modify amino acids in BuS1 was initiated. Different amounts of hydrogen peroxide were added to a standard volume of BuS1 stock solution samples. The treated samples were heated or stored at room temperature. No precipitates were formed in any of the treated samples with or without heat. Eight model fillers and a control have been generated.

Partially reconstituted burley S1 was applied to bright base web without spraying. The solubles and base web were mixed. Preliminary analytical hot water solubles (HWS) data show the percent solids in treated filler from this method of application is lower than HWS from sprayed filler samples. A direct comparison of methods was initiated.

Burley CEL was centrifuged to produce burley solubles (S1) and insolubles.

C. Plans: Submit IT CSC samples from the silver nitrate, hollow fiber membrane, and hydrogen peroxide experiments for S/M assay testing. Freeze dry burley S1 and insolubles and prepare for spraying onto bright base web.

D. References:

1. Izac, R. PM Notebook 8874, pp. 48-55.

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2. Drew, S. PM Notebook 8950, pp. 13-23.
3. Hellams, R. PM Notebook 8959, p. 62.

II. SUPPORT FUNCTION: SAMPLE PREPARATION

A. **Objective:** To prepare chemical fractions and/or condensates as needed for biological and chemical analysis.

B. **Results:** Eleven requests for handmade cigarettes (a total of 647 cigarettes) were completed. Cigarette smoke condensate was prepared for Projects 6912, 6906, 1620, and 2501. A total of 30 samples was smoked. Impaction trap cigarette smoke condensate from two types of cigarettes were collected in quadruplicate.

C. **References:**

1. McGee, N. Personal communication.
2. Hellams, R. PM Notebook, 8959, p 62.

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PROJECT NUMBER: 0008
PROJECT TITLE: Computer Applications Division
WRITTEN BY: John Blankinship, John Palesis, Mark Allred
PERIOD COVERED: May, 1990

I. NEUROCOMPUTING

A. **Objective:** Develop programming techniques for use of the HNC ANZA Plus Neurocomputer and Multilayer Backpropagation Neurosoftware. Investigate potential applications of neurocomputing to R&D problems, and apply where appropriate.

B. **Results:** A new backpropagation neural network was developed for predicting liking scores from analytical data for menthol cigarettes. This network was trained on a training set of 1473 examples from both 85 mm and 100 mm smoking panels. The network incorporates 13 input variables - tar/puff, nicotine/puff, tipping, circumference, menthol/puff, and length for both the test cigarette and the smoker's preferred brand of choice, plus an additional variable for the type of 7-point liking scale. The standard error of the estimate is 0.43 on a 7-point liking scale. Based on predictions against a set of separate test data, the standard error of prediction is 0.49 on a 7-point liking scale.

The general purpose software tool, MBPN Tool, was significantly enhanced to improve the convergence of backpropagation learning. Principal enhancements include: (1) acceleration of learning rates on flat error surfaces, (2) adaptation of momentum terms for more stable and responsive learning, (3) reordering of examples between learning cycles to help avoid local minima, and (4) easier specification of training and testing data sets. These improvements should result in significantly faster convergence and fewer problems with local minima.

C. **Plans:** Using the new MBPN Tool, develop a new backpropagation network for predicting liking scores from analytical data for menthol cigarettes. Use this new network to perform sensitivity analyses to determine the effects of tar, nicotine/puff, menthol/puff, tipping, circumference, and length on liking.

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II. CIGARETTE INFORMATION EXPERT

A. **Objective:** Develop expert system for statistical analysis and interpretation of domestic cigarette information.

B. **Results:** The Cigarette Information Expert was prepared for delivery using the Nexpert Object Runtime Software.

C. **Plans:** Complete delivery of the Cigarette Information Expert. The software will reside and execute on a Sun Server, and will be accessed by personnel in CTSD through VT220 terminals.

III. EXPERT SYSTEMS DEVELOPMENT

A. **Objective:** Develop an Expert System for Cigarette Design.

B. **Results:** We have begun work on the third phase of our Cigarette Design Expert System (CigDES). The primary objective during this phase is to implement a knowledge-based ("heuristic") goal-directed reasoning capability. This new feature will provide a mechanism for resolving the "combinatorial explosion" problems characteristic of the "exhaustive" goal-directed reasoning process already implemented. The main component of the knowledge-based feature is a heuristic function which draws upon qualitative knowledge of cigarette performance to determine what design changes are most likely to lead to a cigarette prototype whose performance satisfies a set of user-defined goals. An initial algorithm for this heuristic function has been designed.

C. **Plans:** We will initially implement a heuristic function which selects cigarette design changes based only on causal relationships between design parameters and cigarette performance. We will then evaluate the heuristic goal-directed reasoning component of CigDES and, if its performance is not satisfactory, we will expand the qualitative model to provide more types of knowledge to the heuristic function.

IV GAS PHASE FIVE-PORT SMOKERS

A. **Objective:** Develop a new data acquisition system (DAS) for the five-port smokers in the CTSD) gas phase laboratory.

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B. **Results:** The Laser Precision Analytical FTIR instrument developed by Kent Koller has been moved to the CTSD Gas Phase Laboratory. Supplementary software for data computations and reporting for the CO, NO, Acetaldehydes, and HCN analyses has been completed and tested . The software for coordinating the operation of the Gas Phase five-port smoker in conjunction with the Laser Precision Analytical data acquisition and processing software is also completed and tested.

The software required to acquire and process Technicon Autoanalyzer Aldehyde and HCN data is being modified to improve the peak detection algorithm for Autoanalyzer data.

C. **Plans:** Software will be completed to support CO, NO, aldehyde, and HCN analyses by the autoanalyzer and FTIR technology.

D. **References:** CAD Project Request #3979

V HP GC CHEMSTATION UPGRADE FOR CTSD

A. **Objective:** Add software and hardware to the HP GC Chemstation to support bar code readers, setup for batchwise processing, post-run reporting and transfer of results to the CTSD LIMS database.

B. **Results:** Programming is complete. The software has been installed on the HP GC Chemstation and is undergoing evaluation.

C. **Plans:** Close out the project. Migrate the programs to other HP GC Chemstations as required.

D. **References:** CAD Project Request #4229

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PROJECT NUMBER: 8101
PROJECT TITLE: Cigarette Testing Services
PROJECT LEADER: Richard A. Thesing
PERIOD COVERED: May, 1990

I. MARKET ACTIVITY

A. Objective: To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

B. Results:

Domestic

Philip Morris has introduced **Bristol Full Flavor 85 and 100, Bristol Ultra Lights 100 and Bristol 85 (Nonfilter)** cigarettes into limited national distribution. These cigarettes will compete against Pyramid and, therefore, will be priced at the subgeneric price level. The **Bristol Ultra Lights** cigarettes deliver 6 mg tar and 0.4 mg nicotine; the **Bristol Full Flavor** cigarettes deliver 16 mg tar and 1.1 mg nicotine; the **Bristol Nonfilter** cigarettes deliver 25 mg tar and 1.6 mg nicotine.

Philip Morris is test marketing a new version of **Next De-Nic® 85 and 100 (plain and menthol)** cigarettes in Tampa, Florida. A major portion of the nicotine has been extracted using the FreePLUS™ system. These cigarettes deliver 9 mg tar and 0.8 mg nicotine.

American Tobacco Company has changed the name of Pall Mall 100 to Pall Mall Gold 100. New package graphics were observed on **Pall Mall Gold 100** cigarettes. The new package is brighter gold in color. No changes were noted in cigarette construction or smoke deliveries.

International - Japan

Select Special Lights King Size 20's (Soft Pack) cigarettes were introduced by Japan Tobacco Inc., to the Japanese market in April. This cigarette delivers 10 mg tar and 0.9 mg nicotine. This product has a dual carbon CA/CA filter.

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II. FIRMNESS

A. **Objective:** To investigate the effect heat sources have on cigarette firmness.

B. **Results:** New procedures that either eliminate or reduce heat sources have been implemented. A semi-automatic temperature probe designed by C. Higgins is currently being evaluated. With this instrument, the temperature of the tobacco bed can now be recorded. A collaborative test between R&D and FTR is planned.

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Program Name	Project Number	Page
International Product Development	4022	19
Low Tar/High Flavor	2307	12
	2304/4015	16
Materials Evaluation*	1759	53
Menthol	2108	1
	1503	29
	1806	30
	1759	53
Microbiological Quality Improvements*	1902	55
New Expansion Processes	1812	36
	1708	46
Optical Processing.	1702	42
Paper Technology.	4009	14
	1720	48
Process Control Systems	5001	22
	1503	29
Project Ambrosia.	2108	1
	2307	12
	4009	14
	2304/4015	16
	1503	29
	6505	76
Project ART	2305	6
	2306	9
	1307	24
	1810	32
	1620	40
	1708	46
	1752	50
	2500	60
	2520	66
Project Natural	2306	9
	2307	12
Project Tomorrow.	1308	26
Project Warhol*	1806	30

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Program Name	Project Number	Page
Reduced Sidestream	4009	14
	1720	48
Selective Filtration	2501	63
Selective Separations	1810	32
	2500	60
	2520	66
Supercritical Fluids	1307	24
	1704	45
	1752	50
Thermal Flavor-Release	2501	63
	2525	68
TSNA	6908	83

*Operations Support

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